# METALLURGIA

THE BRITISH JOURNAL OF METALS

Vol. 64 No. 383

SEPTEMBER, 1961

Monthly: Two Shillings and Sixpence

# NEWTON CHAMBERS

how manufacture and install
BLAST FURNACES and all
ANCILLARY EQUIPMENT
to the designs of JOHN MOHR

Newton Chambers are pleased to announce that they are now sole manufacturers and suppliers within the United Kingdom and elsewhere of complete blast furnaces to the latest designs of John Mohr & Sons, Chicago, U.S.A. Our traditional experience in this field is combined with that of John Mohr & Sons who have been particularly successful in the development of the modern blast furnace for high top pressure.

Consequently, we are now in a position to undertake complete blast furnace installations of the most up-to-date design, to reconstruct and modernise existing blast furnaces, and to offer the whole range of modern blast furnace ancillary equipment.



No. 3 Blast Furnace—Dominion Foundries & Steel LTS Hamilton, Ontario, Canada, designed and installed by John Mohr & Sons. Recent orders include a 28 ft. dia. hearth furnace for United States Steel Corporation at Duquesne Works, to work at 30 lb. per square inch top pressure.

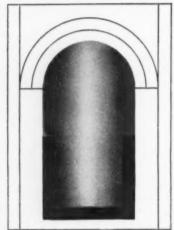


REMEMBER THE EVER INCREASING SCOPE OF NEWTON CHAMBERS
NEWTON CHAMBERS & COMPANY LIMITED, ENGINEERING DIVISION, THORNCLIFFE, SHEFFIELD

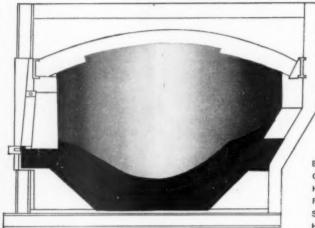
'341' DOLOMITE bricks are manufactured from a dolomitic limestone which in the natural state approaches in chemical composition the mineral dolomite CaCO<sub>3</sub>, M<sub>2</sub>CO<sub>3</sub>, The raw dolomite is dead-burned at high temperatures and it is then formed into suitable shapes, by a new and unique brick-making process. Tunnel kiln firing at high temperatures produces a dense. volume stable and truly basic refractory of exceptional quality. '341' DOLOMITE bricks are resistant to hydration and they can be transported and stocked in the same way as other basic bricks. '341' DOLOMITE bricks are manufactured in various sizes designed to suit many applications.

### DOLOMITE BRICKS

#### for BASIC OPEN HEARTH FURNACES



BASIC OPEN HEARTH SLAG POCKET



BASIC OPEN HEARTH FURNACE SUB HEARTH

#### '341' DOLOMITE BRICKS RAMMED DOLOMITE

'341' DOLOMITE bricks have proved to be ideal for use in modern O.H. units, particularly in sub hearths and slag pockets. In slag pockets these bricks contain the slag during operation - but form a means of speeding up slag removal at the end of the campaign. ('341' bricks hydrate with water to disrupt

and break up the slag.)

#### **Typical Chemical Analysis**

SiO <sub>3</sub>	TiO <sub>2</sub>	Fe <sub>2</sub> O <sub>3</sub>	MnO	Al <sub>2</sub> O <sub>3</sub>	CaO	MgO
2/3%	0.2%	1/2%	0.1%	1/2%	48/50%	36/40%

#### **Typical Physical Properties**

TRUE POROSITY %	18/20	SPALLING INDEX	Cycles +30
BULK DENSITY grams per c.c.	2.70/2.75	(small prism test) PERMANENT LINEAR CH.	ANGE
SPECIFIC GRAVITY	3.35	2 hrs. at 1,500°C.	Nil to 0.2%
SPECIFIC HEAT	0.25	WEIGHT PER CUBIC FOO	T 175 lbs.
PERMEABILITY c.g.s. units	0.05	WEIGHT PER CUBIC METE	RE 2,700 kilos.
COLD CRUSHING STRENGTH p.s Kilos sq.cm.	s.i. +8,500 + 600	9" x 4\frac{1}{2}" x 3" appro	-

**GENEFAX GROUP** for

Everything in Refractories

Please contact our Technical Service Department for further particulars of these and other applications for G.R. '341' bricks.

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Furnaces for annealing, normalizing, bright annealing, carburizing, hardening and tempering, forging, re-heating, malleablizing, cyanide hardening, bright brazing.

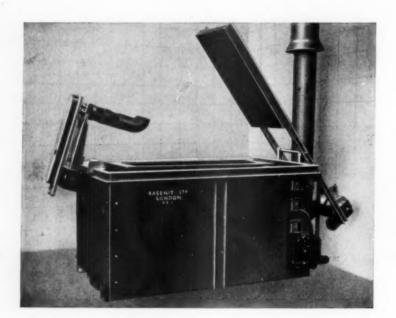
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NATURAL DRAUGHT GAS FIRED MAR TEMPERING SALT BATH WITH AGITATOR Size of bath 36' long × 24' wide × 18' deep

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#### **Thermocouple Wires**

#### THE FACTS

T1\* and T2\* are thermocouple wires of the nickel-chromium/nickel-aluminium type. They are suitable for constant use at temperatures up to 1100°C, and for spot readings at higher temperatures.

T1\* and T2\* conform to the relevant British Standard and Air Ministry specifications. They are entirely British made. They are protected by registered trade marks.

So much for the basic facts. T1\* and T2\* are thermocouple alloys whose stability, life, and quality generally are taken as read by so many pyrometer manufacturers. But these alloys also pass with flying colours the kind of tests by which a first-class commercial proposition is judged.

Made in Britain The commercial advantages of material manufactured entirely in this country are perhaps self-evident. Lines of communication are short and to the point. We can have a finger on the pulse of the market. And, most important of all, we can give an all-round service worthy of products which are very good indeed.

**Small Quantities** — Because of the special nature of the thermocouple market, we are geared to supplying as little as a few ounces of T1\* and T2\*, if that is all you need.

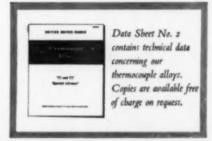
 And Large Which is not to say that we aren't fully able to cope with orders in the upper poundage bracket.

Wire or Strip Both are in constant production in a range of standard sizes (and their metric equivalents).

'Specials' Non-standard sizes or unusual profiles can be supplied.

**Delivery** Both T1\* and T2\* are manufactured in this country and are in daily production. Consequently there are clear advantages in delivery.

**Technical Advice** A well-informed objective opinion on a thermocouple problem may well be worth having. Free and without obligation, of course. \*Regd. Trade Mark





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Furnace

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Could be? Temperatures slightly above optimum may have little or no adverse effect on the plant, the process or the product but there can be no argument about their effect on the balance sheet. Fuel costs money whether it's used efficiently or wasted.

On the other hand, running at slightly below optimum doesn't usually save money. 'Rejects' are also costly—if only in time and labour. Whichever way you look at it there's a case for treating optimum as optimum...a strong case for really accurate, consistently accurate temperature measurement and control. That's West's business on five continents and there's always a West man not so very far, away ready to show you how well he knows his business.

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OA/7285

# America, Pickford Holland are now making to-day's most successful basic roof keys.

The P.H. Ferrobond CMX 3" x 3" Roof Key is a new, metal-clad, chemically bonded basic open-hearth brick without internal plates, designed by E. J. Lavino & Co. of Philadelphia for maximum service and economy. A series of comparative tests with internally plated 4½" x 3" keys proved Ferrobond to be always equal, and usually superior, in performance. They are light and easy to handle, have built-in hot face expansion, and tabs ensure correct alignment. In addition, saving on material cost by rib and valley construction makes the P.H. Ferrobond beyond doubt today's outstanding value in basic roof brick. Considering a trial? Send drawings of your present roof to Pickford Holland who will redesign to suit Ferrobond bricks, and give you the assistance of a service engineer for installation.



#### See how split-roof tests prove OUTSTANDING PERFORMANCE by new FERROBOND ROOF KEYS

	-		Total		% AI	Doygan	
Furnace Capacity	Construction of Roof	Campaign Life	Total Tonnage Produced	Tons/ Hour	Lavino Forrobond CMX 3" x 3" Keys	hond Internally Reinforced 3" Keys 4½" x 3" Keys	Lances through Roof
190 Tens	12" Rib 9" Valley	264 Heats 112 Days	54,000 Tons	28	1.0%	1.6%	2
190 Tens	12" Rib 9" Valley	250 Heats 75 Days	47,500 Tons	26	2.2%	12.6%	2
190 Tons	All 12"	405 Heats 130 Bays	77,000 Tons	25	62%	67%	2
200 Tons	15" Rib 12" Valley	488 Heats 196 Bays	100,000 Tons	21	2.8%	6.6%	MONE
345 Tons	15" Rib 12" Valley	453 Heats 170 Bays	157,000 Tons	38.5	10.4%	% 19.6%	
400 Tons	15" Rib 12" Valley	298 Heats 186 Bays	120,000 Tons	27	NIL	NIL	2

This chart shows comparative performance records in North America of six typical open hearth roofs. In each case the roof was constructed half-andhalf of Lavino Ferrobond CMX 3" x 3" Roof Keys and Internally Plated 4½" x 3" Roof Keys, All were of the Fairless type roof construction (hold-up, hold-down). Production rate of tons per hour is based on total production over campaign time, and includes repair times, fettling times, and all lost time whatever the cause.

P.H. Ferrobond Roof Keys are manufactured under licence from E. J. Lavino & Company, Philadelphia, U.S.A. by

#### PICKFORD HOLLAND

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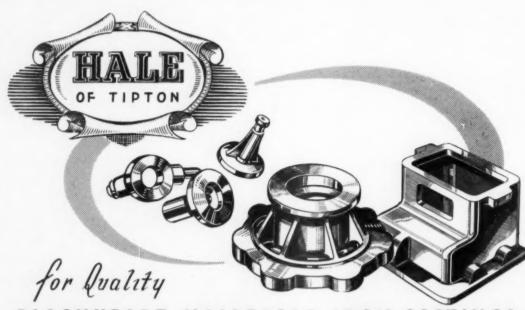
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			Methods of Use	Refrac- toriness	Thermal	Warinen	Approx, weight required	
	Susplied				Hardening Temperature	Temperature of use	cubic foot lbs.	gubic metre kgs.
Maksiccar Plastic	Plastic	Heat Setting	Rand	1730°C	800°C	1580°C	149	2243
Stein Sillimanite Palch	Plastic		maulding		1000°C	1760°C	155	2483
Stein 73 Plastic	Plastic		patching	+ 1758°C	1888°C	1880°C	188	2884
Maksiccar Fire Pulty	Plastic	Air Setting	Nand moulding	1350°C	500°C	1100°C	149	2243
Stein Chrome Patch	Semi- Plastic		or patching	+ 1750°C	880°C	1500°C	200	3284
Maksiccar Patch	Plastic Semi- Plastic Fry			1699°C	0°969	1558°C	135	2162
Stein 73 Patch			Ramming	+ 1750°C	806°C	1800°C	175	2804
Stein Magnesite Ramming Mixture			patching	+ 1750°C	1400°C	1750°C	175	2894

For further details of the full range of Plastic Refractories, Refractory Cements and Castable Refractories, ask for Pamphlet No. 4.

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\* REFRAX is a registered trade mark of THE CARBORUNDUM COMPANY LIMITED

- Will withstand temperatures above 1800°C.
- Very high hot strength combined with great resistance to thermal shock.
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Some Industrial uses

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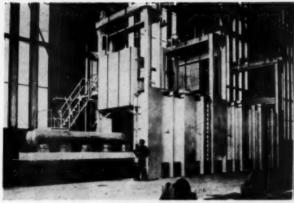
A platinum metal may be the cheapest answer to a heat or corrosion problem.

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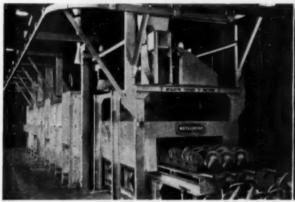
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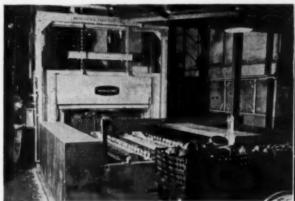
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(RIGHT) Inconel alloy furnace racks in use. (LEFT) Inconel is also used for mesh baskets like this, in which small components are immersed for treatment in a salt-bath. Salt-bath furnace electrodes are also of Inconel.

Design engineers and others interested are invited to-

SEND FOR THIS PUBLICATION

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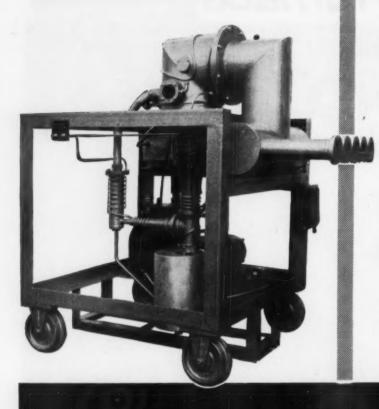
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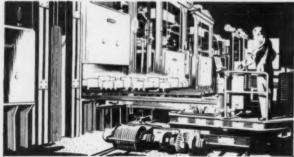
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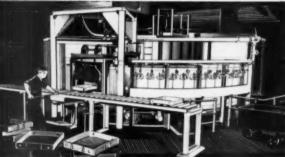
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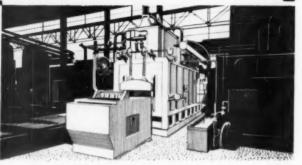
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GIBBONS PRO-TEK Continuous Billet Heating Furnaces ensure scale-free high quality finish

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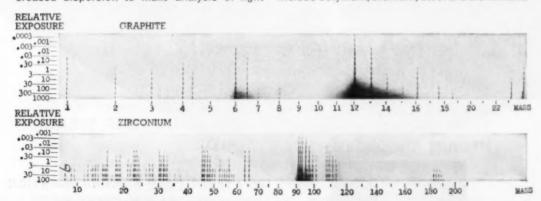
Exceptionally high sensitivity (limits down to .001 p.p.m. atomic) and simple spectrum.

Linearity of ion current with concentration.

The spectra shown are reproduction of plates obtained for analysis of zirconium and graphite. The graphite spectrum illustrates use of increased dispersion to make analysis of light



elements easier and more accurate. Other materials used in reactor technology have been analysed by the MS7 mass spectrometer and include beryllium, uranium, steel and aluminium.



Technical Information Sheet A2 give detection limits for impurities in graphite and Sheet A14 limits for impurities in aluminium.

Figures are given for 72 elements in aluminium; 15 have detection limits of .001 p.p.m. (atomic) or less, 38 limits between .002 and .01 p.p.m. and 19 limits between .02 and .1 p.p.m.

Technical Information sheets on this and other subjects are available on request to:

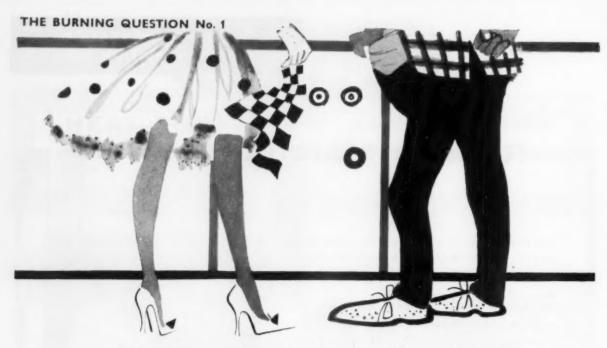
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'Case hardened' is the term.

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GENERAL HEAT TREATMENT, PROCESS HEATING, TESTING, FORGING, METAL MELTING, GALVANISING, TINNING. You name it, Brayshaw will supply the complete answer. Write today for full details of the entire range of BRAYSHAW furnaces, Gas, Oil or Electric—for ALL industrial heat treatments.

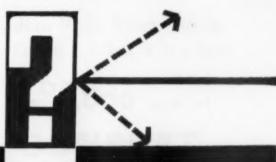
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For operating temperatures of 650/1200°C. Extensively used for hardening of carbon steels, normalising, annealing, pre-heating, case-hardening and general heat treatment.

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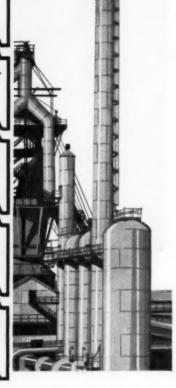
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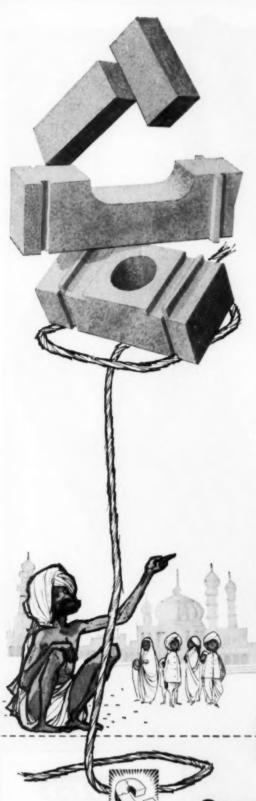


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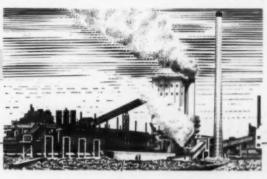
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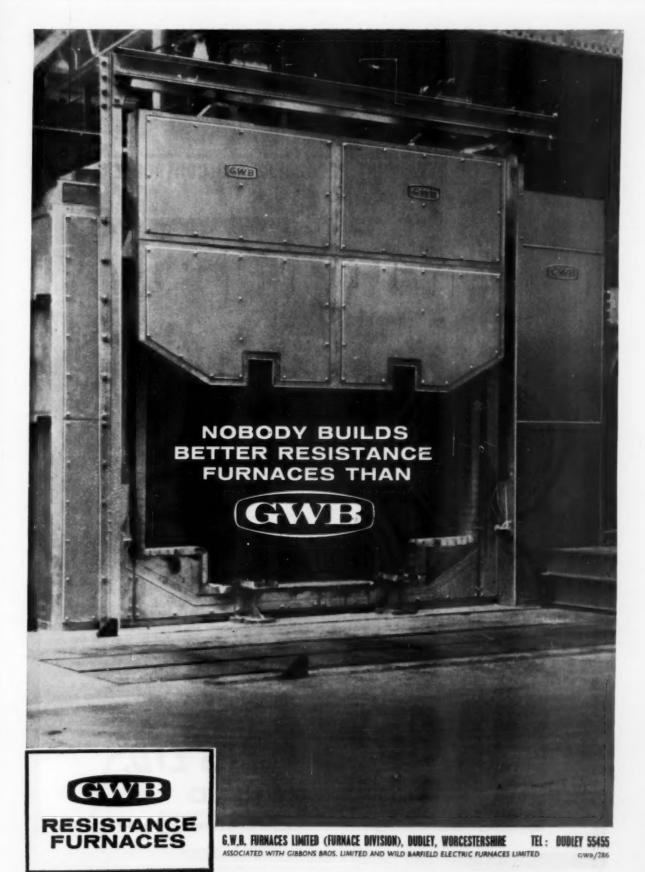
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29

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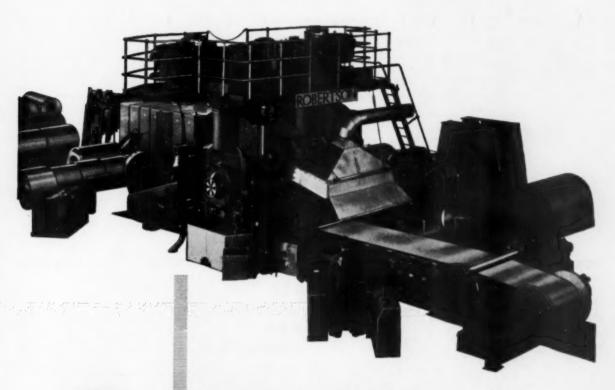


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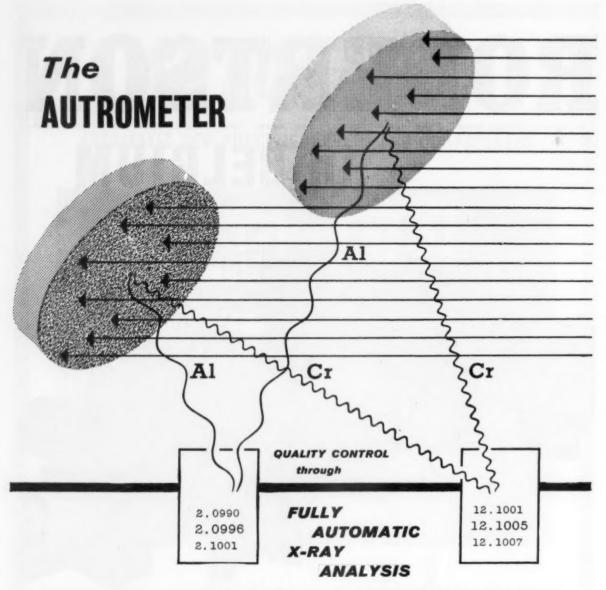
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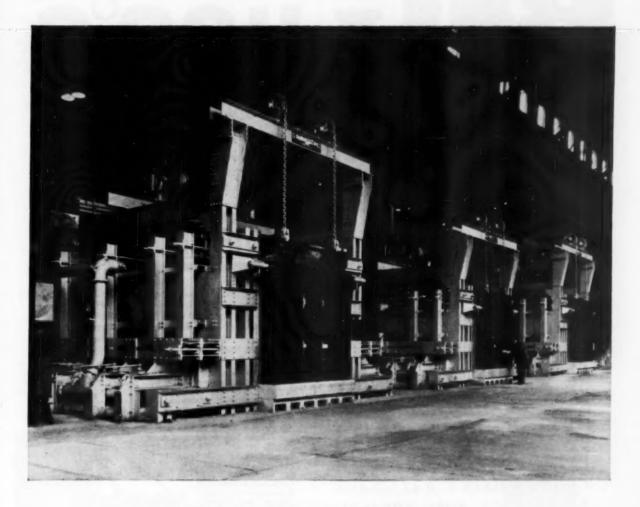
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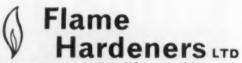


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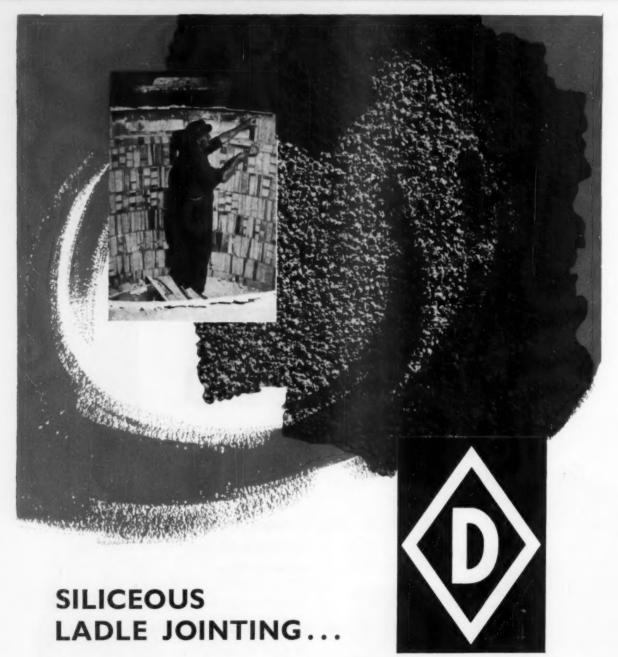
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# METALLURGIA

# THE BRITISH JOURNAL OF METALS INCORPORATING THE METALLURGICAL ENGINEER

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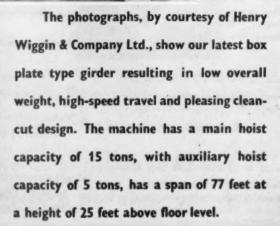
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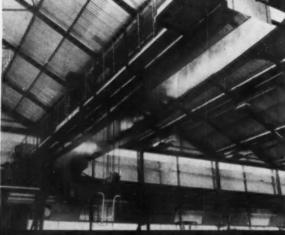
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# METALLURGIA

THE BRITISH JOURNAL OF METALS

SEPTEMBER, 1961

Vol. LXIV. No. 383

### The Reluctant Exporter

THROUGHOUT the post-war period there has been an export drive of sorts. Sometimes it has been pursued vigorously, at others the progress has been about as rapid as that of a motor car on the Exeter by-pass on a summer Saturday. The fact that a country such as ours—with a high population density and a scarcity of minerals—needs to export a not inconsiderable proportion of its industrial output is now widely accepted. Everybody—or almost everybody—agrees on this point, but it is surprising how many believe exporting to be something which concerns "the other chap," with the result that the volume of our exports is much smaller than it could or should be.

It is not possible to attribute to a single factor the failure of firms to export. In an attempt to throw some light on the subject, Marplan, Ltd., on behalf of the Institute of Directors' Export Action Now Committee, has recently carried out a survey of company attitudes towards exporting. Fifty-two firms were questioned—all with less than three hundred employees—the survey being confined to smaller firms because it was felt that it was among these that the greatest expansion of exports was possible.

"Even bearing in mind the modest and restricted scale of this pilot study, one major finding emerges beyond all possible doubt," says the report on the survey. "In the overwhelming majority of cases, the failure of the British manufacturer to increase or even maintain his exports, or to get into the export market at all, is a function not of any external circumstances but of his general attitude towards exports. It is not that he cannot export, but simply that he does not want to."

Of the firms surveyed, about 25% were exporting vigorously; the exports of 50% were declining or stationary at a low level; and just under 25% had never exported and were not really interest in doing so. Less than 10% of the non-exporting firms wanted to export but did not know how to set about it. Among the reasons, or should it be excuses, put forward to account for failure to export, the difficulty of getting hold of adequate information appeared frequently. In this connection a remarkable fact brought to light by the survey was the difference of opinion between managing directors of exporting companies and those of nonexporting companies. The former found that they could easily get export information; that the Board of Trade was helpful; and that lack of foreign languages was no barrier to trade. Many non-exporters, however, gave these reasons as the prime cause for not entering the markets abroad.

An important practical contribution in this field takes the form of the Export Assistance Register recently published by the Export Action Now Committee.

It has been compiled from answers to a questionnaire sent to the 37,000 members of the Institute of Directors asking if their companies needed help to begin selling overseas or, alternatively, were able and prepared to give it. Listed in the Register are some 600 companiesmanufacturing more than 500 different main categories of goods-who have offered export assistance to noncompeting concerns. These companies have undertaken to give free export help and guidance of a general nature, although specific propositions will be a matter for individual arrangement between the companies concerned. "We believe the Register will be most valuable for the potential exporter." said Mr. C. O. Stanley. chairman of the Export Action Now Committee. "When we have assessed results and additional offers of help. we can decide whether the scheme can be extended, for future editions, outside the Institute's membership to industry as a whole. Meanwhile, copies of the first edition are available to interested trade associations on

Industry generally prides itself on having a more progressive outlook than the civil service, but in this matter of exporting many heads of industrial concerns seem to possess a "civil servant" mentality, if we take as our definition that given by Sir Ben Lockspeiser in a speech at a luncheon some years ago when he described a civil servant as "one who finds a difficulty for every solution." So it is with some potential exporters—as soon as you enlighten them on the matter of obtaining information and guidance they raise objections on other grounds. This is only natural when, as the report says, a conspicuously low proportion of managing directors were really enthusiastic about, or even interested in, this side of their business.

Most manufacturers interviewed agreed that a high level of home market demand deterred them from exploring export possibilities, the attitude being that when home demand fell off would be the time to look at the possibility of exporting. Not only does such a view ignore the fact that a product selling well here may be quite unsuitable for overseas markets, it also indicates a failure to realise that any really major reduction in home demand would almost certainly be the consequence of a world recession which might well close up the possibility of exports.

From a study of the comments recorded in the report, it would seem that many of the manufacturers interviewed tend to lack confidence in their ability to capture and sell in overseas markets. In a number of respects they feel their firms are too small to adapt their goods for foreign markets, or to employ an export manager—or even a clerk—to deal efficiently with their export trade, or to allow the managing director to travel abroad and make the necessary contacts. "However," says the report, "this lack of confidence has spread further. A fair number of the manufacturers interviewed are

diffident about the success of the export drive, and look on competitors such as Germany not with any idea of emulation but with resignation at their seizure of former British markets. Their attitude to new overseas markets is not adventurous, and many enquiries which come to the firm are ignored, or summarily dismissed as impossible. They are easily scared off by an order or a contact that goes wrong, and prefer the safety of the home market. However, not only in their export trade but also in the whole of their operations there are signs of a lack of drive, interest, or imagination, and their

knowledge of economic issues or the possible use of marketing and research facilities is remarkably limited."

The main hope in the export field remains the larger firms, but it is clear from the survey that a large number of firms working in the home market at a reasonable level of efficiency could well improve their own position by proper attention to exports. In fact, there is ample evidence in the report that the self-interest of the individual manufacturer and the long-term interests of national economy can both be served by greater attention to the possibilities of export.

#### **New Process Finned Tube**

Over 600,000 ft. of welded finned tube made by a new process is to be supplied by the Extended Surface Tube Co. Ltd., Broad Street Chambers, Birmingham, for use in the boilers of Dungeness nuclear power station. The company is jointly owned by Imperial Chemical Industries, Ltd., and Stewards and Lloyds, Ltd., and the finning machines used for the order were designed and manufactured by Stewarts and Lloyds at their Corby works.

In the new process, using welding current generators among the most powerful of their type, cold rolled strip is welded to the tube as it is helically wound on to form the fin. As a result of strict control, a weld bead is produced which is small and consistent in quality and allows more fins per inch. Up to seven fins per inch are accepted as standard, probably the highest number ever produced in welded fin tube. The process gives good physical properties and allows tight radius bends.

The size range of the tubes produced will complement the company's Integron finned tube, which is made by a rotary extrusion process and of which more than 2 million ft. have been used in the boilers of the Bradwell and Berkeley nuclear power stations and in the Tokai-Mura Japanese nuclear power station.

The fact that the tube is made in the Extended Surface Tube Company's new factory at Corby, adjoining the integrated steel and tube works of the parent company, makes possible a chain of control that is probably unique for this type of product. Control can be exercised from the raw material stage (the actual mining of the ore) to the finished tube.

An interesting feature of the Dungeness order is that some of the tubes for high pressure duty will be supplied in 27 ton minimum tensile steel to B.S. 3059/13 and it is believed that this is the first time that this material has been supplied in the form of welded fin tube. The low pressure tubes will be made from 20 ton minimum tensile material to B.S. 3059/3. All are electric resistance weld tubes.

#### Carburised Rock Drilling Rods

A NEW company has been set up to produce, for the first time in this country, fully carburised rods for rock drilling. The name of the company is Bedford Rock Drill Components, Ltd., and it is to begin its life on a site adjacent to the main Lion Works of John Bedford & Sons, Ltd., the old-established Sheffield firm of steel manufacturers; henceforth the two companies will work in close association. Plant for the new firm is now approaching completion and it is hoped to begin produc-

tion during September. Bedford Rock Drill Components represent only the fourth concern in the world to undertake the production of these fully carburised rods, the three existing plants being in Denver, U.S.A., Johannesburg and Canada.

The reasons behind the decision to set up this new company are to be found in the development, over recent years, of the rock drilling process known as "long hole drilling." This involves using heavy specialised drilling machines, frequently drilling holes of 80 or 100 ft. in depth, for which sectional drill steels are required. These drills are made up of a tungsten carbide detachable bit with screw thread connection as the cutting end, a series of sectional rods which screw into a coupling, and a shank piece.

The pioneers in the development of this type of equipment were the Gardner-Denver Company of Quincy, Illinois and Denver, Colarado, who use a carburising process which gives deep case hardening of the outer skin of the extension rods and shanks and internal case hardening of the hole down the centre of the bar. The couplings are case hardened on a similar principle.

This carburising process, requiring specialised furnaces of unusual design and considerable know-how, is now to be undertaken by Bedford, under licence from Gardner Denver. The patented Hi-Leed thread, which has been developed by the same American company for the threaded ends of the rods and couplings, will also be used at the Sheffield works. Although the most obvious use of the carburising process is on extension rods and couplings, the results of tests are said to have shown that the performances of drill rods for use with tapered and screw thread bits of various types treated by this method is far in advance of the conventional drill steels at present manufactured.

As soon as production of these rods begins it is expected that there will be substantial sales for export through leading rock drilling machinery manufacturers in Britain and overseas.

WILD-BARFIELD ELECTRIC FURNACES, LTD., of Watford, Herts., have received an order from the Austin Motor Company for the supply of a number of electric furnaces for hardening and refining shackle spring pins and other parts at the new B.M.C. works at Bathgate. The installation will comprise a PCF.2 (H) pan conveyor furnace; two standard gas carburising furnaces, model GC.3624, with the carburising atmosphere derived from Carbodrip; and a forced air circulation furnace, model 3660C, for tempering and any other work requiring a maximum operating temperature of 700° C.

# Metallurgical Aspects of the Production of High-Strength Rocket Motor Cases

By P. F. Langstone, B.Sc., A.I.M.

Bristol Aerojets, Ltd.

This article summarises present knowledge concerning metallurgical aspects of the production of high strength rocket motor cases. Methods of improving motor case strengths without incurring weight penalties or loss in reliability by specifying steels having optimum alloy additions and low impurity levels, and by correct choice of fabrication techniques, are discussed.

EVELOPMENT investigations into the production, processing and properties of common and exotic metals are increasing in number and extent but, of the basic materials available in quantity at the present time, only two are of major importance in rocket motor case manufacture; these are low alloy steels and titanium alloys. The considerable progress in the development of high strength titanium alloys in recent years has induced the steel producers to carry out intensive research into methods of improving the strength and ductility of alloy steels. This competition ensures the production of high strength materials and is, therefore, beneficial to motor case manufacturers.

Solid fuel rockets consist basically of a cylindrical case containing the fuel, an igniter assembly, and an exhaust nozzle. The majority of motor cases are fabricated from sheet, rolled into a tube and welded longitudinally, and head and rear end forgings which are connected to the tube by circumferential welding. Thrust take-off and location brackets may be welded to

weld joint alignment are important factors if the material is to be used at its full potential.

The requirement to minimise weight without reduction in strength or stiffness has necessitated the fabrication

the chamber or end forgings. Good weldability and exact

in strength or stiffness has necessitated the fabrication of complex structures incorporating honeycomb or waffle patterns. Integral structures, due to the uniform transference of service loads, are stronger than riveted or welded components. Techniques have been developed for the chemical contouring of motor cases to produce integral waffle patterns.

Motor cases heat treated to material strength levels greater than 90 tons/sq. in. have, on ultimate pressure test, produced erratic results and, in some instances, premature failure at stress levels considerably below the tensile strength of parent metal test specimens heat treated with the component. Premature failure during bi-axial stressing is considered to be due primarily to notch sensitivity caused by lack of adequate surface ductility. In order to ensure the production of consistently high strength motor cases it is necessary to reduce the material notch sensitivity and to devise laboratory tests capable of correlating test piece and component results.

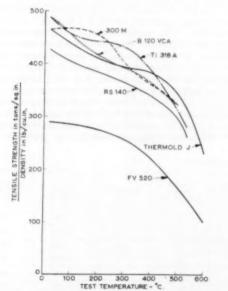


Fig. 1.—Tensile strength to weight ratio of high strength steels and titanium alloys.

#### **Motor Case Materials**

Materials used in the manufacture of rocket motor cases must fulfil a number of structural requirements, including:

- (1) high strength to weight ratio,
- (2) high stiffness to weight ratio,
- (3) availability as sheet and forgings,
- (4) ease of fabrication.
- (5) reliability in component form, and
- (6) low cost.

The tensile strength to weight ratios of four steels and two titanium alloys at ambient and elevated temperatures are shown in Fig. 1, and the chemical compositions and tensile properties are detailed in Tables I and II. It is apparent from the strength/weight ratio results that at ambient temperature the Ti.318 A and B.120 VCA titanium alloys, at tensile strengths of 78 and 85 tons/sq. in., respectively, have approximately a 4% advantage over steels heat treated to a tensile strength of 130 tons/sq. in.

						Averag	ze Composit	ion (%)					
	A1	V	Cr	Ti	C	Mn	8i	8	P	Ni	Mo	Cu	Fe
Fi.318 A	6-9	4-0	-	Rem.	-	-	-	-	-	-	_	-	
B.120 VCA	8-0	13-0	11-0	Rem.	-	-	-	m-100	-	-	-	-	-
M 000		0.08	0.95		0-40	0-76	1.60	-	-0000	2.0	0.50	_	Rem.
RS.140	00	0-20	3-0	-	0.46	0-65	0.20	0.010 max.	0.015 max.	0.25	1.0	-	Rem.
Thermold J		1.0	5.0		0.53	0.35	1.1	-	-	1.5	1:4		Rem.
V 520		-	17-0	_	0.05	1.0	-	_	_	5-0	2.0	2.0	Rem.

Titanium alloys are readily weldable but stringent precautions are necessary during the welding operation to avoid embrittlement by dissolved gases. Trace quantities of oxides, nitrides and hydrides are sufficient to impair the ductility seriously. By maintaining adequate gas shielding with a trailing argon shroud and argon backing, however, it is practicable to weld titanium alloys without a sealed chamber.

Hartbower and Orner¹ have shown that high strength titanium alloys are subject to notch sensitivity and that there is a marked difference in results between different casts. They also noted directionality effects in the sheet. The enlarged grain size in welds and immediate heat-affected zones increases the notch sensitivity. In consequence, welding in titanium development motor cases is usually restricted to circumferential joints, the tube being formed by flow turning. A further effect of the large grain size, reported by Sohn,² is in the response of the weld to ageing. After ageing, the weld strength is greater than that of the small grain size parent material and there is a corresponding loss in ductility. In the majority of titanium alloy development motor cases the thickness of the parent metal in the region of welds is increased to allow for the reduced ductility.

Ageing times after solutionising must be prolonged—up to 72 hours for the B.120 VCA alloy—if the full mechanical properties are to be obtained. As the ageing response is influenced by prior cold work, the ageing time for achievement of optimum properties being reduced by cold deformation, present practice is to age cold worked material. As it is impossible to ensure that all items of a motor case have received the same degree of cold work, variations in properties throughout the case are un-

The cost of titanium alloys is a serious disadvantage. Mid 1960 prices, detailed in Table III, show the cost of titanium alloy sheet to be between 40 and 45 times that for the same gauge of 3% chromium-molybdenum-

vanadium steel sheet. The trend however, is for a narrowing of the gap between titanium and steel prices.

Steels are employed in the majority of motor cases primarily because of the availability of material, the experience already gained in processing techniques and inspection procedures, the wealth of knowledge concerning properties, and the low cost. Titanium alloys have an increased strength to weight efficiency but the major barriers to their greater usage at present are notch sensitivity of welds, high cost, and unknown reliability. Compromises to a limited extent in weight and cost are permissible, but no reduction in reliability can be tolerated. Experimentation with titanium alloys, however, is beneficial in providing knowledge of material properties and in permitting familiarisation with fabrication techniques.

Problems associated with the manufacture of high strength, low alloy steel motor cases have received intensive study during recent years. The following sections of this paper indicate the type of composition required to provide high strength properties, and detail the methods adopted to overcome the difficulties which initially prevented the attainment of consistently high strengths in steel motor cases.

#### Alloy Additions in High Strength Steels

The strength of low alloy steels is mainly governed by the martensitic hardness, which is a function of the carbon content. The tensile strength increases with increasing carbon content, the strengthening effect tending to decrease at higher tempering temperatures. Increments in the carbon content above 0.40%, however, impair the ductility, toughness and weldability, unless the excess carbon is in combination with elements which form carbides. It is generally accepted that to obtain minimum notch sensitivity the carbon content should be as low as possible consistent with the desired strength level. However, Shannon and his co-workers<sup>3</sup> have

TABLE II.-MATERIAL PROPERTIES

Material	Supplier	Tempering or Ageing Temperature (° C.)	0.2% Proof Stress (tons/sq. in.)	Tensile Strength (tons/sq. in.)	Elongation (%)	Density (lb./cu. in.)	Strength to Density Ratio
Ti.318 A	Imperial Chemical Industries Ltd.	540	73	78	12	0.161	485
B.120 VCA	Crucible Steel Co.	480	78	85	9	0.175	485
300 M	International Nickel Co.	320	110	130	9	0-281	463
RS.140	Bristol Aerojet Ltd.	450	90	108	7	0.281	384
	Specification	300	95	120	6	0.281	428
Thermold J	Crucible Steel Co.	320	108	130	9	0.280	465
FV 520	Pirth Vickers Ltd.	450	72	80	9	0.277	290

#### TABLE III.—COST OF SHEET MATERIAL (mid. 1960)

					Sheet Cos	t (per lb.)
					16 8.W.G.	18 S.W.G.
Ti.318 A	 	 	 	 	£ s. d.	£ s. d. 8 7 0
B.120 VCA		 	 	 	6 12 0	-
RS.140	 	 	 	 	0 3 2	0 3 7

shown that for H 11. 5% chromium steel, the carbon content should not be below 0.35% for minimum notch sensitivity at any given strength level.

The primary function of alloying elements is to increase the hardenability and matrix strength. Chromium and manganese increase the solid solution hardening and retard softening on tempering. Nickel strengthens the ferrite, improves the hardenability and lowers the impact transition temperature. Silicon raises the temperature of martensite breakdown thereby increasing the resistance to tempering. Vanadium, molybdenum and titanium promote secondary hardening by the formation of complex carbides.

Rocket motor steels can be divided into three broad classifications:—

- (1) Martensitic steels tempered at a temperature sufficiently high to reduce residual stresses but below the temperature for precipitation of cementite or alloy carbides along the martensite plates. Tempering is carried out in the region of 200° C., which is below the ductility trough, the tensile strengths attained being over 100 tons/sq. in.
- (2) Martensitic steels modified by the addition of 1·5-2% silicon, thus enabling tempering at a higher temperature without decrease in strength. These steels develop tensile strengths of over 100 tons/sq. in. when tempered at 300-350° C. The silicon addition, however, increases the danger of inclusions. Surface and sub-surface inclusions are particularly undesirable in steels heat treated to high strength levels.
- (3) Secondary hardening steels, in which high strength is attained by precipitation of complex carbides during tempering. The secondary hardening characteristics depend on the type and amount of alloy addition. Strong secondary hardening steels can develop tensile strengths in excess of 120 tons/sq. in. when tempered as high as 550° C. For full hardening complete solution of all carbides is necessary prior to quenching. With a high alloy content this entails an elevated solutionising

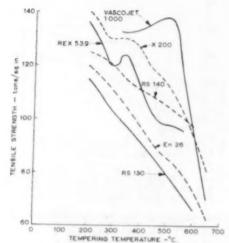


Fig. 2.—Response of high strength steels to tempering. temperature which may have attendant difficulties due to excessive decarburisation or dis-

The chemical compositions of steels typical of these three groups are detailed in Table IV and strength property/tempering temperature curves are shown in Fig. 2. Secondary hardening steels are capable of achieving the highest strength levels combined with good ductility, and increased application of this type is to be expected.

#### Weldability

Motor case welds must be of the highest quality. High impurity content materials are directly responsible for hot and cold cracking, and incorrect welding techniques for weld misalignment and peaking, all of which are possible causes of premature failure.

Hot and cold cracking can occur in both the weld metal and heat-affected zone. Wilkinson and Cottrell<sup>4</sup> have reported the effect of sulphur in promoting hot cracking, strains imposed during cooling causing rupture of the sulphur-rich, low-melting-point, grain boundary constituent. This detrimental effect is greater with higher carbon steels as, during solidification, carbon competes with sulphur for sites within the grain, the excess sulphur being rejected to the boundaries. Phosphorus also is an undesirable element due to its effect of increasing the segregation of sulphur and carbon. Other elements such as arsenic, antimony, tin, lead and

TABLE IV.—HIGH STRENGTH STEELS

Type	Steel	Supplier		1	Average	Compo	eition (	%)		Tempering Temperature	Tensile
13/60	ctee	cultimer	C	Mn	Ni	Or	Mo	V	81	(° C.)	Strength (tons/sq. in.
1	SAE 4240 RS 130° En 26	=	0-40 0-32 0-40	0-78 0-50 0-60	1 · 88 0 · 20 2 · 5	0-80 1-0 0-65	0·25 0·20 0·55	111	0·25 0·20 0·20	200 200 200	195 115 120
9	Rex 529 Super Hytuf Airsteel X200	Thos. Firth & John Beown Ltd. Crucible Steel Co. United States Steel Co.	0-35 0-40 0-43	1:57 1:30 0:85	1.8	0-11 1-4 2-1	0.34 0.35 0.55	0·21 0·20 0·06	1 · 55 2 · 30 1 · 55	300 300 300	190 120 120
(medium Mo, and V)	NCMV R8,140°	English Steel Corporation, Lt.1.	0-45	0-80 0-65	1·7 0·25	1·45 8·0	1.0	0-24	0-90	300 300	136 120
(high Mo and V)	HST 140 Vascojet 1000	Samuel Pox & Co. Ltd. Vanadium Alloya Steel Co.	0-40	0-80	=	5-0 5-0	2·0 1·3	0-45	0-90	600 530	136 120

<sup>° 8, 0.010</sup> max.; P, 0.015 max. (Bristol Aerojet, Ltd., specification)

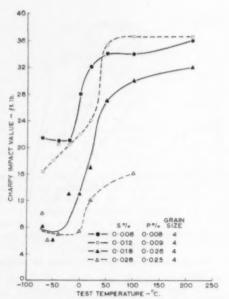


Fig. 3.—Effect of sulphur and phosphorus contents on Charpy impact value.

bismuth may form low melting point constituents which, at grain boundaries, would increase the tendency for hot cracking. Further work is required to confirm the

effect of these impurities.

Manganese combines with sulphur to form a relatively high melting point constituent and has, therefore, a beneficial effect. The carbide formers are also beneficial, probably, as suggested by Lukashevich-Duvanova, due to the formation of stable compounds of chromium, molybdenum and vanadium with sulphur and carbon. It is assumed that when sulphides have formed stable compounds with carbides, the sulphur has no deleterious effect on the steel.

Cold cracking in the heat-affected zone is caused by a combination of hydrogen, cooling rate and stress imposed by restraint. Berry and Allan suggest that hydrogen promotes crack propagation as soon as an embryo crack has formed. The initiation point can be a discontinuity such as an inclusion, a grain boundary film, or a small pre-existing hot crack. Cooling rate and restraint stresses also promote crack propagation due to their effect on austenite producing a critical mixture of high and low temperature decomposition products. The interdependent variables have an additive effect on cold cracking.

It is essential, therefore, that steels selected for motor case manufacture are of high cleanliness and are low in impurity elements, especially sulphur, phosphorus and hydrogen.

#### **Brittle Fracture**

Motor cases made from steels heat treated to high strength levels have, in many instances, failed on test at a stress considerably below the material tensile strength. Failures at low stresses have presented two problems—to obviate brittle fracture and to devise a laboratory test which will ensure correlation of test specimen and component results.

Brittle fractures are caused by notch sensitivity, a peculiarity that is particularly evident in steels at high strength levels. The fracture plane of brittle failures is generally perpendicular to the sheet surface and there is negligible plastic deformation. The fracture surface is crystalline, although there may be narrow shear lips along the sheet surfaces. The notch ductility at strength levels below 80–100 tons/sq. in. is, in general, sufficiently high to tolerate minor stress concentrations.

There are two sources of weakness—stress concentrations, and chemical agents in grain boundaries or on crack faces that lower the surface energy of the material. The theoretical aspects of this subject have been dis-

cussed by Cottrell.7

All fractures, whether ductile or brittle, develop in two stages—crack initiation and crack propagation. Every motor case, however carefully constructed, contains points of high stress concentration, and in localised areas the yield stress may be exceeded and cracks initiated at a low overall load. The source of the localised stress concentration may be a mechanically induced notch, a sharp change in section, a weld defect, or a flaw in the material. If the localised stresses are accommodated by slip, the stress concentration at the tip of the crack is relieved by the movement of dislocations in adjoining grains and the crack is arrested. If slip is restricted. however, the stress at the tip of the crack builds up, causing the crack to extend until it reaches the critical size at which it propagates rapidly, resulting in brittle fracture.

For any specific flaw size there is a fracture toughness value that corresponds to the maximum usable strength. The optimum strength condition is the one in which failure by general yielding and by crack propagation are

equally probable.

Any factor which restricts plastic flow increases the tendency towards brittle fracture. An increase in yield stress retards plastic flow; a rise in yield stress alone, however, does not necessarily imply brittleness but, in the presence of small quantities of impurities which also hinder slip, it can lead to catastrophic failure. Both a decrease in tempering temperature, which increases the yield stress, and the addition of carbon and alloying elements that raise the yield point without reducing the grain size, can result in brittle behaviour.

Precipitates, inclusions and foreign atoms are all agents which hinder plastic flow and reduce the material ductility. Sulphur, in particular, in addition to promoting weld hot cracking, adversely affects the steel tough-The impact strengths of 75 tons/sq. in. 1% chromium-molybdenum steels (SAE 4130) with various sulphur and phosphorus levels are shown in Fig. 3. A reduction in grain size acts in a manner similar to a decrease in sulphur content. This effect, shown in Fig. 4. is expected, a small grain size material having an increased grain boundary area and thus a thinner and less continuous film of detrimental sulphides at the boundaries. A small grain size may also affect the propagation of fracture due to the number of boundaries at which changes in direction take place in the passage of the fracture from one grain of differing orientation to the next. A report, by Hodge et al8 confirms the deleterious effect of sulphur on impact resistance, the material in this investigation being a 2½% nickel-chromium-molybdenum Low strength, grain boundary impurities also reduce the bend ductility of steel specimens. The

results of bend tests, giving a degree of biaxial stressing, are detailed in Fig. 5 which shows the decrease in ductility as the sulphur and phosphorus contents increase.

Several investigations have been carried out into the susceptibility of high strength steels to hydrogen embrittlement. Delayed brittle failures are associated with the presence of hydrogen, the fracture stress being influenced by the notch acuity and by the amount and distribution of hydrogen. Morlet and his co-workers9 consider that embrittlement results from hydrogen in solution, the hydrogen contained in internal voids being non-damaging, and that in the presence of stress concentrations hydrogen will diffuse to the point of maximum triaxiality of stress. As the detrimental effects occur primarily in the presence of complex stresses and decrease with increasing strain rate, the study of hydrogen embrittlement is best carried out by sustained load tests on notched specimens. Work by Goldhoff et al10 has shown that transverse specimens are more susceptible to hydrogen embrittlement than those taken longitudinally. As mentioned previously, Berry and Allan6 suggest that hydrogen promotes crack propagation from embryo cracks, such as inclusions. The size. distribution, shape and amount of non-metallic inclusions, therefore, are important factors. Inclusions in transverse specimens are more likely to be normal to the stress direction, thus providing areas of high stress concentration leading to easier crack initiation. Goldhoff<sup>10</sup> also states that vacuum melted steel is susceptible to hydrogen embrittlement, but less so than air melted steel. This lower susceptibility may be partially due to the decreased quantity of inclusions in vacuum melted steel.

As the test temperature is lowered the stress needed to produce plastic flow is raised. The ductile fracture stress increases with decreasing temperature until the mode of failure changes from shear to cleavage when the energy required for fracture drops rapidly over a small temperature range. This is called the transition tempera-

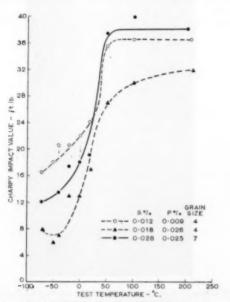


Fig. 4.-Effect of grain size on Charpy impact value.

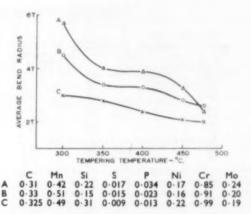


Fig. 5.—Effect of sulphur and phosphorus contents on the ductility of 1% Gr-Mo steel.

ture range. It is preferable that the transition temperature of the material specified for any component should be below the minimum temperature at which the component is stressed during service.

At very high rates of strain slip cannot propagate as fast as the tip of the crack. Therefore, the results of fast strain rate tests, such as the notched impact test, do not bear a direct relationship to the behaviour of components under stress. The impact test, however, despite limitations, can be used to obtain significant data.

It is of interest to note that contributary causes of brittle fracture, high sulphur and hydrogen contents and stress concentrators such as non-metallic inclusions, are also factors which result in poor weldability.

#### **Decarburisation to Combat Brittle Fracture**

It has been established that a partially decarburised skin on high material strength motor case surfaces results in an increase in burst strength, due to the increased surface ductility and corresponding reduction in susceptibility to notch sensitivity. Unless there are flaws in the centre of the parent material or weld, a partially decarburised surface skin will reduce any tendency towards brittle behaviour. Any crack initiating at a surface stress concentrator is arrested due to plastic flow in the ductile surface layer. Stress concentrations producing local stresses above the material yield stress are considered necessary for the crack to propagate into the brittle material in the sheet centre.

The surfaces of motor cases made by Bristol Aerojet, Ltd., are partially decarburised by use of a controlledcarbon-potential, endothermic gas atmosphere during heat treatment. Results of burst tests on oil quenched and tempered RS 140 steel motor cases and tubes are detailed in Table V, together with values for the depth of partial decarburisation. A motor case at the moment of failure is illustrated in Fig. 6. A distinction is made between motor cases, which consist of tubes with end forgings attached by welding, and tubes tested without end rings. In pressurised tubes circumferential or hoop tension stresses are predominant, whereas in motor cases longitudinal stresses are also present. Where the longitudinal stress is half the hoop stress, the theoretical value for a perfect case, the shear strain theory of von Mises-Hencky claims that in elastic failure the hoop

		Tomorelea	1	Material Properties		Depth	Hoop	Hoop Stress to
Serial No.	Component	Temperature (° C.)	0·1% Proof Stress (tons/sq. in.)	Tensile Strength (tons/sq. in.)	Elongation (%)	Decarburisation (in.)	Stress (tons/sq. in.)	Tensile Strength Ratio
DMT 188	Tube	450	83 · 8	104 · 8	6 · 5	0-001	98 · 0	0-94
189		450	84 · 5	108 · 1	6 · 5	None	84 · 0	0-78
193		450	82 · 0	106 · 3	7 · 5	0-003	106 · 0	1-00
194		450	82 · 3	107 · 8	8 · 0	0-0025	103 · 0	0-96
195 196 197 198	99 99 99	450 450 450 450	85 · 7 84 · 5 82 · 5 85 · 0	111 · 0 109 · 2 107 · 0 107 · 9	7·3 8·0 6·5 5·3	0 · 002 0 · 003 0 · 0015 0 · 0015	106 · 8 107 · 8 110 · 7 113 · 5	0·96 0·99 1·04 1·05
199	0 p	450	82·7	108 · 1	7·5	0 · 003	110 · 7	1.03
300	0 p	450	85·3	110 · 7	7·0	0 · 002	108 · 7	0.98
201	2 p	450	84·9	110 · 4	7·0	0 · 0025	104 · 7	0.94
195	Case	450	81-5	102 · 8	8·5	None	79 · 5	0 · 77
197		450	82-6	104 · 4	8·5	0-001	107 · 0	1 · 02
142		850	65-5	92 · 6	10·5	0-0015	102 · 0	1 · 10
143	00	450	76-3	97·3	7·5	0-005	106-5	1 · 09
174	00	550	71-0	101·3	9·0	0-008	115-0	1 · 14
175	00	500	77-5	102·0	9·5	0-0035	105-5	1 · 03
176	29	550	70 - 7	99 · 4	9·5	0·004	115 · 5	1 · 16
179		500	82 - 6	106 · 1	9·5	None	70 · 8	0 · 66
WLT 31		450	86 - 8	107 · 5	8·0	None	86 · 5	0 · 80

stress to material strength ratio should be approximately 1·15:1. Thus, providing that stress concentrations are avoided, higher hoop stresses are expected for cases than for tubes.

The degree of surface decarburisation was estimated from the results of micro-hardness traverses across sections of the tubes, the depth of partial decarburisation quoted in Table V being the distance from the surface to a hardness reading 50 points D.P.N. below the core hardness.

It is obvious from the graph relating hoop-stress/material-tensile-strength ratio to depth of decarburisation (Fig. 7) that partial decarburisation to a minimum depth of 0·0015-0·002 in. is beneficial in obviating brittle fracture. The maximum permissible depth of decarburisation has not been determined, but it must bear some relationship to the tube wall thickness.

Shank and his co-workers<sup>11</sup> have also found that deliberately decarburising to a depth of 0·003–0·005 in. provides greater surface toughness, and thereby reduces the danger of brittle fracture. They report the history of a motor case which failed prematurely, the fracture initiating at an area where a protrusion, formed during flow turning, was removed by grinding. The protrusion was ground after heat treatment, surface decarburisation at this area being removed. Although the tube contained a 0·006 in. deep lap, this defect did not initiate a crack. It should be noted that the surfaces of the lap were decarburised. It is inferred that small defects are less dangerous when left in the steel than when removed by grinding after decarburisation of the surface.

Surface decarburisation reduces the fatigue properties of steels heat treated to tensile strengths less than 90 tons/sq. in., but its effect on the fatigue life of ultra high strength steels has not received full investigation. Where fatigue strength is the criterion, surface decarburisation, although beneficial in reducing notch sensitivity, should be avoided until its effect has been fully investigated.

#### **Laboratory Tests**

The motor case hoop strength is an indication of the ability to resist biaxial stresses and, at strength levels

over approximately 90 tons/sq. in., it bears no relationship to the uniaxial tensile strength of the material.

Several types of test have been developed for evaluating the fracture resistance characteristics of sheet metals. Although considerably more experience is needed in correlating laboratory tests with the performance of motor cases, crack propagation tests can differentiate between steels of similar composition and



Fig 6.—Motor tube at instant of failure: arrow indicates point of initiation of failure.

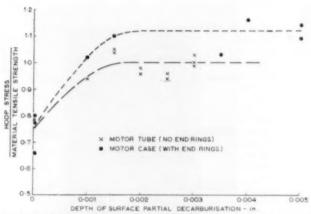


Fig. 7—Relationship between hoop stress/tensile strength ratio and surface decarburisation (oil quenched and tempered condition).

tensile strength but having differing brittle behaviour and can also evaluate the effect of processing variables. The tests give an indication of the potential of new materials under development and serve as quality control tests for production materials. Two tests which are frequently used are the notch tensile and the instrumented bend tests. As the fracture stress decreases markedly at temperatures below the transition range, it is necessary for crack propagation tests to be conducted at or below the anticipated minimum service temperature.

#### Notch Tensile Test

Details regarding the design, preparation and testing procedure for the sharp-edge notch tensile specimen recommended by the A.S.T.M. have been published recently.<sup>12</sup> The specimen dimensions are shown in Fig. 8. The criterion for notch sensitivity is based on the ratio of notch strength to tensile strength. Up to a strength level in the region of 90 tons/sq. in., the notch tensile

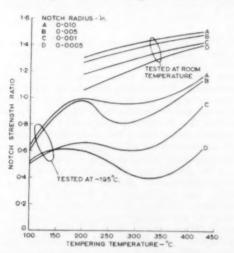


Fig. 9.—Notch strength properties of S.A.E. 4,340 steel for various stress concentrations.<sup>13</sup>

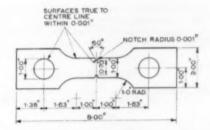


Fig. 8.—A.S.T.M. notch tensile test specimen.

strength of sharp notched specimens from sheet material is equal to the tensile strength, but above about 90 tons/sq. in. the notch tensile strength is a measure of the notch ductility and the theoretical maximum load may not be attained. The sharp drop in notch strength at higher strength levels is related to the enhanced crack propagation as a result of the decrease in ductility

in the presence of the stress concentration provided by the notch.

The effect of varying the notch radius, thereby producing differing stress concentrations, has been shown by Sachs et al.<sup>13</sup> Results of tensile tests carried out on notched round bar specimens at room temperature and —195° C. are reproduced in Fig. 9. The increased notch sensitivity caused by decreasing notch radii is readily seen from these results. Klier<sup>14</sup> and his co-workers show that at strength levels above 80 tons/sq. in., the impact test is a less sensitive index of the fracture characteristics of the steel than is the notch tensile test. The results of this work, reproduced in Fig. 10, indicate that more impact than notch tensile tests would be required to establish a comparable precision of testing for a high strength steel.

#### Instrumented Bend Test

In the Allison instrumented bend test a small unnotched rectangular specimen, 0.052 in.  $\times$  0.700 in.  $\times$  1.5 in., is biaxially stressed to failure by bending. The specimen is readily machined and can easily be obtained, if required, from motor cases after burst test. During bend test the rate of deflection is maintained constant and the load on the specimen, determined by a resistance

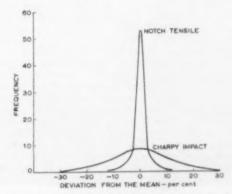


Fig. 10.—Comparison of distribution curves for impact and notch tensile tests.<sup>14</sup>

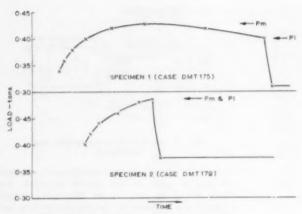


Fig. 11.—Instrumented bend test results.

strain gauge load cell, is recorded as a load-time curve. Typical curves for specimens sectioned from two high strength steel rocket motor cases are shown in Fig. 11. The load increases gradually to a maximum (Pm) and then falls to a certain load (P1) at which the specimen fails by rapid crack propagation. The difference between the maximum stress and the stress at rapid failure is a measure of the material's capacity for redistributing high localised stresses and it indicates the susceptibility to brittle failure. Specimen 1 in Fig. 11 was taken from motor case DMT 175 which burst at 105.5 tons/sq. in., and specimen 2 from case DMT 179 which failed at a hoop stress of 70.8 tons/sq. in., the test piece tensile strength to case hoop stress ratios being 1.03 and 0.66, respectively.

#### **Heat Treatment**

Residual stresses play an important role in the fracture of motor cases, quenching and tempering stresses contributing to heterogeneity and unbalanced stress fields under bi-axial test conditions. Micro-stresses may effect nucleation of cracks whilst macro-stresses may increase the rate of crack propagation. Surface tensile stresses arising at changes in section during heat treatment, being additional to the external tensile load, may cause premature failure.

Stresses may be introduced during heat treatment, depending on changes in material section, the nature of the quenching medium, and the transformation characteristics of the steel. Varying section thicknesses are unavoidable in motor cases, but endeavours should be made at the design stage to reduce the severity of sectional changes. High hardenability, air hardening steels are normally chosen for use at high strength levels. Hardening in air, a less drastic operation than oil quenching, reduces residual stresses and distortion of the motor case. The higher the quenching speed the greater the temperature difference between the surface and the centre of the component, the surface transforming to martensite whilst the centre is still austenitic. There is an increase in volume during the austenite to martensite transformation and transformation in stages results in non-uniform expansion causing excessive residual stress.

#### **Fabrication Processes**

Theoretically a seamless case represents the ultimate in reliability and, although it is not practicable to

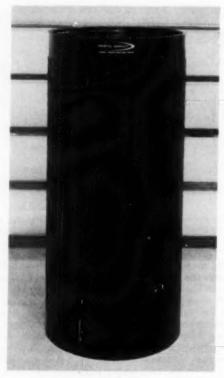


Fig. 12.—Chemically contoured 24 in. diameter motor tube.

achieve this with the present state of technology, seamless tubes with a flange at one end can be produced by flowturning. In addition to a reduction in the number of welds, flowturning enables more accurate control of wall thickness than can be obtained when using sheet steel. Lodge & Shipley, Ltd., have manufactured a flowturning machine capable of producing cylinders up to 80 in. in diameter and 30 ft. in length from one piece of steel

Flowturning, however, has not yet been universally accepted, many manufacturers awaiting further information concerning the reliability of flowturned high tensile strength cases before accepting this method of manufacture. Flowturning disturbs the metal structure to such an extent that the higher impurity content core may subsequently form the surface layers. It is possible, therefore, that flowturned cases may be more susceptible to brittle fracture due to the notch effect at surface laps or at surface and sub-surface non-metallic inclusions.

The extremely critical strength to weight ratio requirements of missiles has necessitated the fabrication of complex structures. Brazed honeycomb tubes show promise but manufacturing difficulties have arisen, particularly with large diameter, thin skin tubes. During heating to the brazing temperature distortion of the skins and of the tube is a problem unless complex jigging is provided. Accurate fit between core and skins and perfect cleanliness are necessary to ensure complete bonding by the brazing alloy. Inspection of the finished honeycomb for completeness of braze has presented

difficulties: methods employed include radiography. ultrasonic inspection and suction cup testing.

For the production of components incorporating integral stiffeners, chemical contouring is unsurpassed. Chemical contouring is the controlled removal of metal from defined areas of a component by chemical attack. Techniques developed for contouring 1% chromiummolybdenum steel have been reported 15 and etchants are now available for steels of other compositions. It is possible to produce very thin lands without mechanical distortion, the etching producing a smooth, blended radius which reduces the stress concentration at sectional changes. A waffle pattern etched by a single process in the exterior surface of a 24 in, diameter tube is illustrated in Fig. 12. Where varying final thicknesses are required the component is etched in several stages, with sequential removal of predetermined areas of the maskant. Tapers can be achieved by a controlled rate of immersion in or withdrawal from the etch solution.

#### Conclusions

In order to achieve the highest possible strengths in steel motor cases it is essential to obviate brittle fracture. It is necessary to specify extremely low impurity steel, to carry out stringent quality control tests on incoming material, to reduce stress concentrations and to increase the material ductility. Every endeavour must be made to eliminate surface and sub-surface defects and to prevent the introduction of hydrogen during welding, pickling and heat treatment. Detailed inspection. especially of welds, is of paramount importance,

Rocket motor case burst strengths in the order of 105 tons/sq. in. can be achieved consistently by specify-

ing low impurity, 3% chromium-molybdenum-vanadium steel and by controlling the carbon potential of the heat treatment furnace atmosphere to produce partial decarburisation of the component surfaces to a minimum depth of 0.002 in.

#### Acknowledgments

The author wishes to thank the directors of Bristol Aerojet, Ltd., for permission to publish this article.

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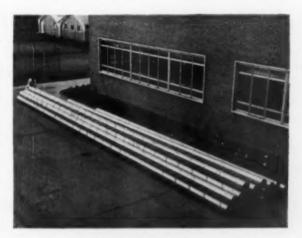
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## Helical-Weld Aluminium Pipe

ARGE-SCALE production of aluminium pipe in sizes up to 28 in. diameter is now possible, due to the successful adaptation to aluminium of the helical-weld method of tube making. Until now the two main types of aluminium tubing that were available in large quantities, extruded and drawn seamless tubing, have been limited by practical considerations to a maximum diameter of approximately 12 in. The method sometimes used to fabricate large diameter pipes, by bending plates into cylindrical shapes and welding them longitudinally, does not lend itself to large-scale production.

The method of making helical-weld tube consists essentially of feeding metal strip into formers or a series of rollers set at an angle so that the strip describes a helix as it travels through the machine. The resulting helical seam is then welded continously and automatically. The tubing can be made in very long lengths and to accurate limits. The process was first developed for steel pipe at the end of the last century but it took many years of improvements in strip quality, in automatic welding techniques, and particularly in continuous weld testing and quality control, before the production of consistently high-quality helical-weld pipe became a practical proposition. Because of the increasing demand for thin-walled aluminium pipe in large diameters, Alcan Industries, Ltd. (then Northern Aluminium Co., Ltd.) undertook in 1959, in collaboration with makers and operators of helical-weld equipment,

a programme of development work on the production of helical-weld aluminium tube. The close tolerances to which aluminium strip is supplied permit welding to take place under excellent edge matching conditions. This is an important point which has contributed significantly to the success in making aluminium helical-



Large diameter thin-walled aluminium pipes made by helical welding.

weld pipe of consistently high quality. Moreover, welding is faster with aluminium than with steel, and the high speeds possible lead to high rate of output and low

manufacturing costs.

Helical-weld tubing is now being offered, in sizes from 6 in. to 28 in. diameter with wall thicknesses up to \$\frac{1}{4}\$ in., by three British companies: Apitubes, Ltd., Bristol Aerojet, Ltd., and The British Steel Piling Co., Ltd. Apitubes are producing pipe on helical-weld equipment designed and built at their Jarrow-on-Tyne works; Bristol Aerojet have developed a different type of machine which produces helical-weld tube to exceptionally close tolerances for the more specialised types of application; and British Steel Piling are operating a Driam helical-weld machine at their Claydon Works, near Ipswich. On the Continent, at Oberhausen, near Düsseldorf, Intercontinental Enterprises G.m.b.H. are building their Heliweld plant. This equipment is capable

of forming tube up to 120 in. diameter from strip up to ½ in. thick; it can therefore be used for making cylindrical vessels and tanks as well as tubing. For all trials on helical-weld machines in this country Alcan Industries, Ltd., advised on machine adaptation and supplied Noral strip up to ½ in. thick in large coils of the higher strength alloys. Their associates, Aluminium Laboratories, Ltd., Banbury, fitted the MIG and TIG welding equipment to the machines and established correct welding procedures.

There are many pipework applications where aluminium has inherent advantages over other materials, but has hitherto been subject to serious size and cost limitations. The introduction of helical-weld aluminium pipe means that a new assessment of the role of aluminium in pipework is possible, because it can now be obtained in a wider range of sizes and often at a lower

cost than that made by other processes.

## New Works for Royce Electric Furnaces

IN a new factory occupying a prominent corner position in the L.C.C. Industrial Development Area at Sheerwater, near Woking in Surrey, Royce Electric Furnaces, Ltd., has been rehoused and re-equipped to cope with the increasing demand for the special furnaces it manufactures. Popular demand is for "packaged units" which do not involve assembly work on site, and most of the Royce furnaces, whether of standard design or made to meet precise requirements, are delivered bricked and wired and needing only connection to an electricity supply to be put into commission.

The new factory has a floor space of about 40,000 sq. ft. and is arranged to provide two main bays, 190 ft. in length, for the manufacturing and assembly work which involves metal cutting and fabrication, laying insulating brickwork and refractories, and electrical installations. A machine shop occupies one side of the factory and on the other side are stores, works offices and canteen.

The fabricating section has been well equipped with machinery for sheet metal working and for rolling angles and channels. A guillotine with a 10 ft. blade can cut steel plate up to ½ in. thick, and a hydraulic bending press is used for streamlined body construction. There is an automatic profiling machine for making intricate shapes, and argon-arc welding equipment has been installed for joining heat-resisting steels. A new feature in the electrical section is a furnace testing panel which enables a comprehensive check to be made on heating uniformity, electrical circuits, controls and insulation. Modern equipment for cutting hard refractories and thermal insulation boards is installed in a dust-proof room.

The products of the factory range from low temperature ovens to furnaces providing temperatures up to 1,750° C., and include equipment for heating materials in air, in gas atmospheres and in vacuum. Furnaces are made for all metal heat treatments and for applications in the glass and pottery industries. They are supplied to users at home and abroad and many are installed in government establishments.

The ability of the company to meet the demand for special types of furnaces depends upon first class design facilities, and co-operation is essential between designers, sales and works engineers. The sales and design staff have ready access to the works since together with the administration staff, they occupy a new office block in front of the factory. Here they work in light and spacious offices with windows overlooking the residential part of the area.

The Honeywell Ultra-Vision flame detector has been reduced from £66 11s. to £45 (list), as a result of increased acceptance of the unit in the United Kingdom. It is now in use in a number of installations in the steel, electricity, petroleum, chemical and other fuel-using industries.

The Manchester office of The David Brown Corporation (Sales), Ltd., is now located at Ashburton Road, Trafford Park, Manchester 17 (Tel: Trafford Park 4751/2), where the company's machine tool division is now established.



A Royce furnace ready to leave the new factory.

# Some Experiments on the Hot-Tinning of Small Parts

By C. J. Thwaites, M.Sc., A.R.S.M., A.I.M.\*

The results are reported of a systematic study of the effect of tinning practice on the thickness and uniformity of coating on small parts tinned by the hot-dip and hot-barrel processes. It is shown that the former, followed by centrifuging, is more universally applicable. An acceptable standard of finish is coincident with a mean coating thickness of 0.0003 in., when 75% of a batch will have a thickness between 0.00025 and 0.00035 in. with none below 0.00015 in.

OATINGS of pure tin or a tin-lead alloy may be applied by immersing suitably prepared small parts, contained in a mesh basket, in a bath of the molten metal, followed by a treatment to prevent the fusing together of individual parts during solidification of the coating. The process is commonly used for components such as tags, terminals, pins and sockets for the radio, electrical and electronic industries. The excellent solderability and protection against corrosion afforded by such coatings have been long recognised. Several papers have appeared in the technical press covering the subject of hot-tinning generally, but not specifically relating to the coating of small parts, 1-6 and, as far as is known, no systematic study has been reported of the effect of tinning practice on the quality of the coating for different types of small wares. The present investigation was therefore aimed at understanding some of the factors affecting the thickness of hot-dipped coatings on small parts and the uniformity of the coating thickness between individuals from a given batch. General appearance of the processed work was also used as a measure of quality, since in commercial practice, this factor may be of considerable importance.

Materials

The parts chosen for the experiments were ? in. No. 6 steel wood-screws. in. steel washers, and four types of brass electrical tags designated Types 1 to 4; these types and the relative sizes may be seen in the illustration, Fig. 1. The screws and washers were chosen to represent, respectively, angular components with rather deep recesses (the thread and the slot in the head) which would tend to trap excessive molten metal by surface tension effects, and parts having completely flat faces which may tend to adhere together strongly. The tags were of

types in common usage in industry and varied with regard to angularity, recesses, apertures or "tubes," and in weight.

#### Coating Techniques

All parts to be processed were degreased in trichlor-ethylene vapour and boiling liquor. The steel screws and washers were pickled in 66 vol.% hydrochloric acid for 10 minutes while the brass tags were immersed for a few seconds in a sulphuric-acid nitric-acid bright-dipping solution. Brass subsequently to be coated with pure tin was barrel-electroplated with a minimum thickness of 0·0001 in. of nickel to avoid contamination of the tinning bath with zinc and copper; this procedure was recommended for all brass work by the Post Office some years ago.  $^7$ 

The prepared parts were transferred to a suitable mesh basket and then immersed for a few seconds in an aqueous flux solution of the following composition:

Zinc chloride					24 lb.
Sodium chloride		0 0		0 0	6 lb.
Ammonium chlor	ide				3 lb.
Hydrochloric acid	1 (8.0	7. 1.	16)		⅓-1 pin
Water to make					10 gal.

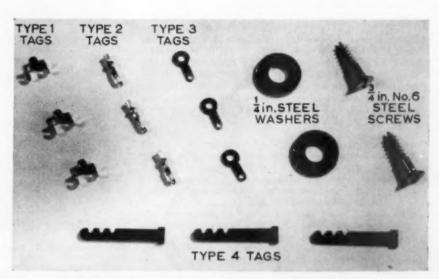


Fig. 1.—Types of small parts used in the investigation.

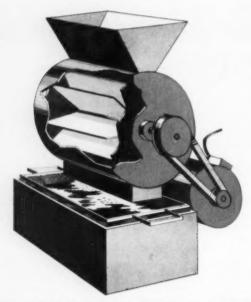


Fig. 2.—Sketch of the impeller-type separator as used at the Tin Research Institute. The circular side plates of the impeller have a minimum clearance (about 0.060 in.) from the sides of the casing. The clearance between the ends of the blades and the casing is 1 in. The impeller is 10 in. in diameter and is driven at between 575 and 1,350 r.p.m.

Application of the coating followed immediately either by hot-dipping or by the hot-barrel method.

For dip-tinning, the work was held in a round, wiremesh basket and immersed for about 10 seconds in the pure tin or the 60% tin-40% lead alloy, both being maintained at  $280^{\circ} \pm 2^{\circ}$  C. The dipping bath had a fused flux blanket partially covering the surface, the flux containing the solid components of the solution given above. The basket was withdrawn from the dipping bath and quickly transferred to a centrifuge. where the parts were spun for periods up to 10 seconds at 1,400 r.p.m. in order to remove excess coating metal. Rotation was stopped by a mechanical brake in less than 0.5 second after the completion of the desired period, and the basket was then removed from the machine. The tinned parts were immediately tipped into the hopper of the separator which was of the paddlewheel type and of 10 in. diameter (Fig. 2). Separator speeds of 575, 725, 950 and 1,350 r.p.m. could be employed. The clearance between the impeller blades and the outer casing (1 in.) was just larger than the largest tag to be tinned. The separated parts passed from an aperture at the bottom of the separator casing into a water-quench tank where they were caught in a wire basket. In a few instances, hot-dipped parts were taken direct from the tinning bath to the separator without intermediate centrifuging.

In the hot-barrel tinning process, a measured weight of the fluxed parts was placed in a hexagonal mild steel barrel which rotated at 20 r.p.m., and which was maintained at a constant temperature by electric heating elements (Fig. 3). In some of the experiments the inner surfaces of the barrel were maintained in a tinned condition, in which case the operating temperature was

controlled at  $300^{\circ}$  C., while in other experiments the steel barrel was initially heavily oxidised and maintained at  $350^{\circ}$  C. Thermocouples inserted into the side and base of the barrel measured the operating temperature. The heaters and thermocouples on the rotating barrel were connected to the control panel and temperature indicator via slip-rings on the barrel spindle and fixed brushes.

In the procedure usually adopted, a weighed load of known surface area was placed in the barrel. When the flux on the work had dried, some tin or tin-lead alloy sheet was placed in the barrel, the weight used being sufficient to give the desired coating thickness. After a suitable period of tumbling, usually between 1.5 and 3 minutes, the parts showed a uniform bright appearance and were then tipped out into the separator. This "end-point" was determined solely by visual appearance, and considerable experience was found to be required to obtain precisely the correct time of treatment.

Fig. 3 also shows the separator placed in position below the hot-barrel apparatus.

#### Coating Thickness Measurements

Twenty individual parts were taken randomly from each batch of tinned articles. The coating thickness was determined by Clarke's gravimetric method<sup>9</sup> on each of 10 individual parts, and again on the remaining 10 taken together. Loss in weight on stripping the coating was converted to mean coating thickness, the density of the coating metal being known, and the surface area of the parts being measured where possible by projection of a magnified silhouette on to a frosted-glass screen.



Fig. 3.—Electrically heated hot-barrel tinning unit with heater control panel and temperature indicator. The barrel rotates at about 20 r.p.m. and normally runs inclined backwards similar to an electroplating barrel. The separator is seen in position below the hot-barrel unit.

In this manner, average coating thickness was determined on 20 parts and some idea of the scatter between individuals obtained from the 10 separate determinations. On two batches only, one of dip-tinned parts and one of hot-barrel-tinned parts, the coating thickness on 50 individual parts was measured in order to plot a coating thickness frequency-distribution curve.

Similar determinations were made on specimens obtained from several commercial tinners in order to compare these with the parts processed in the laboratory.

#### Effect of Tinning Procedure on Mean Coating Thickness

The experimental results from the dip-tinning and hot-barrel-tinning processes will be described separately for convenience. In each case a comparison will be made with commercially produced samples coated by the same method.

#### A-DIP-TINNING

The effects of individual factors during the coating process on mean coating thickness, on variation of coating thickness between individual parts, and on appearance will be described in turn.

#### (i) Centrifuging Time

With constant dipping and separating conditions, the period of centrifuging was varied between 1 and 10 seconds, and the resultant mean coating thickness values are given in Table I. Some values are included for parts receiving no centrifuging prior to separation, these being recorded as zero centrifuging time. These latter results should not be given too much significance, however, since the ultimate thickness would depend, among other factors, on the period elapsing between withdrawal of the work from the dipping bath and its passing through the separator, during which some molten tin could drain off, and on any accidental shaking during this period which would assist in removing the excess metal.

It will be seen that there is a general trend for the coating thickness to decrease with centrifuging time, the most pronounced effect being in the first 5 seconds of spinning. This confirms the previously published results obtained when centrifuging frying pans at 1,325 r.p.m.<sup>10</sup>

The Type 2 tags coated with tin-lead alloy gave mean coating thickness values considerably higher than any of the other parts receiving a similar treatment, although the coating thickness was still dependent on centrifuging time. The differences in thickness of tin and tin-lead alloy coatings are discussed more fully in the section A (iii).

#### (ii) Separating Speed

For a given set of hot-dipping and centrifuging con-

TABLE I.-EFFECT OF CENTRIFUGING TIME ON COATING THICKNESS

Type of	Separator	Mean Coatin	g Thickness (is	n.) for Centrifu	ging Time o
Fmail Part	(r.p.m.)	0 sec.	1 sec.	5 sec.	10 mm.
Screws	575 575** 1350	0·0022 0·00032	0·00036 0·00027	0-00029 0-00024 0-00023	0 · 00025 0 · 00021 0 · 00022
Washers	873 575° 1350	0-00077 0-0004	0·00015 	0-00019 0-00020 0-00017	0.00016 0.00017 0.00016
l'ype 2 Tags	575 575°	=	-	0-00023	0·00021 0·00038

O Coatings of 60% tin-40% lead alloy, the remainder are of pure tin.

ditions, the speed of the separator was varied over the range 575-1.350 r.p.m. The effect on the mean coating thickness and on the tendency for different types of small parts to adhere together by virtue of the surface tension of the molten coating was examined: the results are shown in Table II. It should be noted that separation at any speed other than the highest, without prior centrifuging, generally resulted in excessively thick, non-uniform coatings, and the threads in the screws were partially or completely filled with the coating metal. Separating at 1.350 r.p.m. without centrifuging, however, resulted in much of the excess tin being removed by the impact of the impeller blades, thus giving an acceptable finish. Less than 1% of the washers and Type 3 tags were stuck together after separation at 1.350 r.p.m. without centrifuging, and with screws and angular tags of Type 1 total separation was achieved.

The more angular parts centrifuged before separating gave 100% separation for all separator speeds, but with light flat tags of Type 3, 5 seconds spinning followed by separating at only 575 r.p.m. left up to 30% of the load adhering together, in many cases as clusters of four or five tags.

Increasing the separator speed up to 1,350 r.p.m. was again necessary to achieve total separation with these tags. The coating thickness of parts centrifuged prior to separating was not dependent on separator speed, since there was insufficient thickness of metal remaining to be thrown off during separation.

It was observed that slight damage occurred to sharp edges of parts separated at 1,350 r.p.m., due to the impact with the impeller blades. The damage was rather greater on the heavier parts, such as screws and washers. This defect and means of overcoming it will be discussed later.

#### (iii) Coating Composition

Both pure tin and a 60% tin-40% lead alloy were used for coating certain types of small part under as near as possible identical conditions of dipping, centri-

TABLE II .- EFFECT OF SEPARATOR SPEED ON COATING THICKNESS AND DEGREE OF SEPARATION

					Separator Sp	eed (r.p.m.)			
Type of	Previous	5.7	5	725		950		1,350	
Small Part	Treatment (	Coating Thickness (in.)	Separated (%)	Coating Thickness (in.)	Separated (%)	Coating Thickness (in.)	Separated (%)	Coating Thickness (in.)	Separated
Screws Washers Type 3 Tags Type 3 Tags	No centrifuging No centrifuging 5 secs. centrifuging 5 secs. centrifuging	0 · 0022 0 · 00078 0 · 00029 0 · 00052	95 87 82 70	0-0012 0-00097 0-00028	94 84 97	0.00098 0.00076 0.00023	98 96 93	0 · 00032 0 · 00038 0 · 00024 0 · 00042	100 99 100 99

<sup>°</sup> Coating of 60% tin-40% lead alloy, the rest are pure tin.

TABLE III.—COMPARISON OF THICKNESS OF PURE TIN AND TIN-LEAD TABLE V.—COATING THICKNESS ON COMMERCIALLY COATED TAGS.

Type of	Trea	tment	Mean Coating T	hickness (in.) for
Small Part	Centrifuging Time (sec.)	Separator Speed (r.p.m.)	Pure Tin	00% Tin- 40% Lead
Screws	5 10	575 575	0-00028 0-00025	0-00024 0-00021
Washern	5 10	575 575	0×00019 0×00015	0·00020 0·00017
Type I Tags	5	575	0.00021	0.00057
Type 2 Tags	5 10	575 575	0.00023 0.00021	0 - 00060 0 - 00038
Type 3 Tage	5	575 1,350	0 - 00029 0 - 00024	0-00052 0-00042

fuging and separating. A comparison of the mean coating thicknesses obtained is made in Table III, from which it is clear that there is no consistent difference in thickness of the pure tin and the tin-lead alloy coatings Centrifuged screws and washers carried similar coating thicknesses, whereas all of the tags had tin-lead coatings of greater thickness than those with pure tin coatings. This difference in behaviour cannot be explained by the difference in the viscosities of the two coating metals. since they have rather similar values. Also the tinlead alloy has the lower surface tension value, which would be expected to allow thinner coatings of this metal. It is possible that the presence of lead results in a stronger and more tenacious oxide film enveloping the molten coating, which resists the forces arising during centrifuging.

#### (iv) Tupe of Small Part

In Table IV are shown values of mean coating thickness for different types of component, all having received similar cycles of hot-dipping, centrifuging and separation. There was no great difference in mean thickness for any one processing cycle, although there appeared to be a trend for the flat steel washers to retain the thinnest coating, due, no doubt, to the smooth faces of this component.

#### (v) Commercially Coated Specimens

In Table V are given values of mean coating thickness on different types of commercially tinned tags which are either identical, or closely similar to the patterns used in the laboratory experiments. The details of the tinning procedures are not fully known, but in all except one instance the parts were known to have been centrifuged prior to separating. All these tags were of brass, and it is worth recording that only in three cases was a nickel undercoat applied before hot-dipping. The coating metal was in each case a tin-lead alloy containing either 60 or 70% tin.

A paddle-wheel type separator similar to that in the laboratory was employed by most of these firms, giving

Supplier	Type of Tag	Coating Metal	Mean Coating Thickness (in.
A	3 1 1 1 1 2	70% Sn-30% Pb	0-00040 0-00054 0-00055 0-00064 0-00050 0-9011†
В	4	60% 8n-40% Pb	0-00025†
C	2 2 3	60% 8n-40% Pb	0.00023 0.00022 0.00015
D	2 3	60% Sn-40% Pb	0-00011 0-00021
E	4	60% 8n-40% Pb	0.00022*†
F	3 3	60% Sn-40% Pb	0.00008 0.00012

No centrifuging; separated directly after dipping
 Electroplated nickel undercoat.

a high degree of separation even on flat tags. The one establishment separating by allowing the parts to fall in the centre of a horizontal rotating plate which carried rather closely spaced steel pegs obtained a much lower percentage separation on Type 4 tags, and this confirms laboratory experiments with a machine of this type.

#### (vi) Quality and Coverage of the Coating

Laboratory tinned parts not centrifuged before separating showed an uneven "lumpy" coating, which would not be acceptable commercially, for all separator speeds except the highest, 1,350 r.p.m. At this speed the screws, washers and simple flat tags (Types 3 and 4) had a satisfactory appearance, although with the heavier parts some slight damage occurred on sharp edges due to impact with the separator blades. Tags of Types 1 and 2, not centrifuged, remained partially blocked through the apertures. All parts which had been centrifuged before separating were without exception bright and uniform in appearance; again some slight damage to edges occurred where a high separator speed was used.

All the commercially coated specimens were of satisfactory appearance and free from defects, except for the tags similar to Type 1 from establishment A. Up to one-half of these parts had uncoated patches within the tube-like portion of the tags, despite the mean coating thickness being relatively high.

#### B-Hot-Barrel-Tinning

As mentioned earlier, the barrel was used in both the tinned condition (at 300° C.) and in the untinned and oxidised condition (at 350°C.). Experiments were carried out to determine the range of coating thickness that could be obtained, given acceptable coverage and appearance, by varying the amount of coating metal added to the barrel.

#### (i) Coating Thickness

Table VI summarises the results of these experiments

TABLE IV.—TIN COATING THICKNESS ON SIMILARLY PROCESSED DIFFERENT COMPONENTS

Treat	ment	Mean Tin Coating Thickness (in.) on								
Centrifuging Time (sec.)	Separator Speed (r.p.m.)	Borews	Washers	Type 1 Tags	Type 2 Tags	Type 3 Tag				
5 5 0	575 575 1,330 1,350	0·00028 0·00025 0·00023 0·00032	0.00019 0.00016 0.00017 0.00040	0-00021	0·00023 0·00021	0-00029 0-00024				

Type of Small Part	Condition	Coating		A		ickness (in.) obtained when added to Barrel to give	
Small Part	of Barrel	Metal	0.0001	0.0003	0.0005	0-001	0.002
Type 1 Tags	Oxidised Oxidised Tinned	Pure Tin Tin-Lend Tin-Lend	0-000048 0-0001	Ξ	0-00020 0-00044	0-00038 0-00041 0-00047 0-00037° 0-00032 0-00030 0-00027° 0-00023 0-00030 0-00040°	=
	Oxidised	Tin-Lead (with Ni undercoat)	0.000091	0.00015	0.00021	400	0.00044
Type 2 Tags	Oxidised	Pure Tin	-	0.00020	0.00034	-	-
Screws	Oxidised	Tin-Lead	-	0-00026	_	_	-

<sup>\*</sup> The results of these 2 individual experiments in each case show the reproducibility of coating by this technique.

for different types of work, from which it appears that a substantial proportion of the coating metal is lost in many cases. This "loss" occurred irrespective of whether the barrel was used in the tinned or the oxidised condition. It was not found to be possible to obtain a coating greater than 0.0005 in. in thickness, but this would not normally constitute a serious drawback, since coatings of between 0.00025 and 0.0004 in. thickness are usually adequate. As would be expected, the degree of separation of the coated parts varied in the manner described for hot-dipped and centrifuged components, section A (ii).

#### (ii) Comparison with Commercial Samples

Table VII shows the results of thickness determinations on tags coated commercially by the hot-barrel method. For establishment A, sufficient coating metal was added to the barrel to obtain, in theory, a coating thickness of  $0 \cdot 00084$  in. The amount used by the other plants was not known. In each case the inside of the barrel was tinned and the coating metal was  $60\,\%$  tin– $40\,\%$  lead alloy.

#### (iii) Quality and Coverage of Coating

It was found that the final brightness and uniform appearance of the work depended to a large extent on judging when tinning was complete. To exceed slightly the desired processing time would lead to oxidation and dulling of the work, but insufficient tumbling resulted in many uncoated areas being obtained. An average period of 1.5 minutes was required from the time of adding the metal to the barrel, and the variation in this period was from 1-3 minutes. The condition of the barrel did not appreciably affect tinning time, although there was a tendency for the time to be shorter when using the oxidised barrel at the higher temperature. Coatings of 60% tin-40% lead alloy on brass were often brighter and more pleasing in appearance when the brass was initially electroplated with a 0.0001 in. thick layer of nickel before barrel coating.

The Type 1 and 2 tags tinned by the hot-barrel method, both in the laboratory and commercially, generally had

TABLE VII.—COATING THICKNESS ON COMMERCIALLY HOT-BARREL TINNED TAGS.

Supplier	Type of Small Part				Mean Coating Thicknes (in.)	
A	Tags, similar to type 1				0-00054	
E		9.9	12	9.0	3	0-00015
G		22	-00	22	8	0.00038
H		.00	55	22	4	0.00036

a considerable area of uncoated surface inside the "tube." This defect was eliminated in the laboratory samples by adding enough metal to the barrel to give theoretically a coating of 0·001 in. (resulting in an actual coating 0·0003-0·0005 in. thick). Steel screws were coated satisfactorily and presented a good appearance.

It was found that only small parts which would tumble satisfactorily in the rotating barrel, preferably those rather angular in nature, could be coated to an acceptable standard by the hot-barrel technique. It appeared that the rubbing together of surfaces due to the tumbling action was largely responsible for the uniform distribution of the coating metal. It is therefore not surprising that interior surfaces, as found in tags of Type 2, were difficult to tin, since there was no rubbing action on these surfaces.

#### Variation in Coating Thickness on Laboratory and Commercially Tinned Parts

In order to obtain an indication of the scatter in coating thickness between individual specimens of one treatment, and to observe the effect on this scatter of the factors already discussed, two methods of assessment were employed. Firstly, the proportion of the 10 randomly chosen specimens having coating thickness values lying within  $\pm~0.00005$  in. of the mean value for the batch was noted. This allowed a realistic, but sufficiently close, tolerance for coatings greater than 0.0002 in. in thickness. However, for thinner coatings, for example 0.0001 in., such a tolerance would allow too great a variation about the mean, and a second limit of  $\pm~25\%$  about the mean was therefore employed. It is clear that this latter tolerance would be rather too large for coatings as thick as 0.0004 in., for example.

TABLE VIII.—VARIATION IN COATING THICKNESS ON INDIVIDUAL SCREWS COATED UNDER DIFFERENT CONDITIONS,

Coating Method	Centrifuging Time (sec.)	Separator Speed (r,p,m,)	Mean Couting Thickness (in.)	Percentage within ± 0.00005 in. of Mean	Percentage within ± 25% of Mean
Hot-dipping	0	1,350	0.00027	40	40 90 90 80
	0	1,350	0-00027	80 80	90
	1 1	575	0.00036	60	90
		575	0-90034	90	80
	5	575	0.00025	70	70
	5 =	575	0-00025	100	100
	5 0	575	0.00022	80	80 80
	10	575	0.00026	70	80
	10 0	575	0.00022	60	80
	1 1	1,350	0-00026	60	80
		1,350	0.00025	70	80 20
	10	1,350	0-00022	90	90
Hot-barrel	0	876	0.00026	60	60

Oatings of 60% tin-40% lead alloy; the rest are pure tin.

Table VIII shows the percentage of screws having coating thickness values within the stated limits, for different mean thicknesses and processing conditions. The picture presented by these results was found to apply to every type of small part employed in the investigation and, for a given mean coating thickness and treatment, it appeared that the percentage within the limits varied randomly from 100% down to as low as 20% in certain instances. This variation could not be correlated with mean coating thickness over the approximate range 0.0001-0.0006 in.

Table IX compares the scatter in coating thickness of laboratory and commercially coated electrical tags; each line refers to one processed batch of work. These results show that the random variation in the proportion of articles having coating thicknesses falling between the stated limits, discussed above for laboratory coated work, is also present in commercially processed articles. Indeed, it may be seen that in three batches of Type 3 tags from supplier C, having closely similar mean coating thickness values and, as far as is known, given the same tinning procedure, had in one case 60% and in another 100% of the chosen sample of 10 individual parts within the limits. Table IX also confirms the fact that the scatter does not appear to be related to the coating procedure.

Although laboratory tinned steel washers showed a similar variation in coating thickness between individuals to all the other types of small parts, it was observed that on the individual washers having a thickness falling outside the limits, their divergence from the mean coating thickness was considerably greater than with screws or

From the points of view of corrosion resistance and of solderability, it is usually the few articles in every batch which carry inadequate thickness, or which lack uniformity of coating thickness, that may cause either difficulties in a production process or defects in a finished assembly. Examination of 10 individuals from each batch of coated specimens in the present investigation gave an indication that, for coatings of about 0.0002 in. thickness, the lowest individual thicknesses obtained were about 0.00012–0.00015 in.

However, it was considered desirable to analyse a greater number of individual parts, and 50 parts from each of two batches of screws were therefore taken. each batch having a mean coating thickness value that might be called "desirable," i.e. about 0.0003 in. One batch of screws had been tinned by hot-dipping and centrifuging and the other by the hot-barrel method. It was found that the lowest coating thickness value on any individual screws from these two batches was 0.00017 and 0.00020 in., respectively, for the dipped and the barrel-tinned parts. It is recognised, of course, that such minimum values may be affected to some extent by the type of part being coated or by the precise coating procedure, but it is considered that the experimental results indicate that even the parts having the thinnest coating are adequately coated for the majority of purposes. On the other hand, the highest individual coating thickness values did not exceed 0.0004 in., and would not be expected to lead to tears or build-up of solid tin on sharp edges or in apertures and threads.

#### Discussion of Results

From the experimental work and examination of the commercially coated specimens, it may be concluded

that for any specific type of small part there is a limited range of hot-tinning conditions and techniques which will produce an acceptable standard of finish with regard to uniformity and thickness of coating, coverage by the coating, and a minimum clustering together after the separation process. It would appear that the most suitable conditions for coating the various types of small part employed in this 'investigation may be summarised as follows:—

- (a) Screws: Hot-dip; no centrifuging; separate at 1,350 r.p.m.
  - or Hot-dip; centrifuge 5 sec., 1,400 r.p.m.; separate (speed immaterial within range 575-1.350 r.p.m.).
- or Hot-barrel, aiming for a thickness of at least 0.0003 in.; separate 575-1,350 r.p.m.
- (b) Washers: Similar conditions to those in (a) above.
  (c) Type 1 tags: Hot-dip by either method as for screws in (a)
  - above.

    or Hot-barrel, aiming for a thickness of 0.001 in.; separate 575-1,350 r.p.m.
- in.; separate 575-1,350 r.p.m.
  (d) Type 2 tags: Hot-dip; centrifuge 10 sec., 1,400 r.p.m.; separate 575-1,350 r.p.m.
- (e) Type 3 tags: Hot-dip; centrifuge 5 sec., 1,400 r.p.m.; separate at 1,350 r.p.m.
- (f) Type 4 tags: Hot-dip; no centrifuging; separate at not less than 700 r.p.m.
  - or Hot-dip; centrifuge 5 sec.; separate at not less than 700 r.p.m.

TABLE IX.—COMPARISON OF SCATTER IN COATING THICKNESS ON INDIVIDUAL TAGS COATED COMMERCIALLY AND IN THE LABORATORY.

Coating Conditions	Type of Tag	Supplier	Mean Coating Thickness (in.)	Percentage within ± 0.00005 in. of Mean	Percentage within ± 25% of Mean
Hot-dip:	1	Laboratory	0-00018	60	60
centrifuge :		processed	0-00043	70	90
separate		1	0.00055	50	86
			0.00078	40	90
		Λ	0.00035	40	70
	2	Laboratory	0-00019	70	70
		processed	0.00025	70	70
		D	0-00011	100	100
	3	Laboratory	0.00022	50	50
		processed	0.00042	80	100
		A	0.00039	70	100
		C	0.00015	100	70
			0.00022	60	60
			0.00023	100	100
		D	0.00021	50	50
		P	0.00008	90	50
			0.00012	100	90
	4	В	0.00025	80	80
Hot-dip; separate	1	Laboratory processed	0.00055	40	50
	4	E	0.00022	40	40
Hot-barrel	1	Laboratory	0.00050	100	70
		processed	0.00010	90	80
1			0.00022	90	90
			0.00032	30	60
			0.00037	50	70
			0.00043	20	60
			0-00047	60	90
		A	0.00054	30	90
		G	0-00038	40	70
	2	Laboratory	0.00019	90	90
		processed	0-00033	30	60
		E	0.00015	90	60
	4	H	0.00036	100	100

These conditions apply to coatings of pure tin, or alloys of tin and lead with up to 60% of tin. As a general rule, these optimum tinning conditions result in a mean coating thickness of 0.0002-0.00035 in., a value which agrees well with the recommended minimum thickness for good solderability. 11.12 To exceed a thickness of about 0.0004 in, may result in an irregular surface with blockage of apertures, and external dimensions outside the allowable tolerance. Ultra-thin coatings below 0.0002 in, may have poor appearance and give inadequate protection against corrosion, or may have inferior solderability after storage under unfavourable conditions. For this range of coating thickness, the difference

in mean coating thickness values for batches of parts treated in an identical manner is likely to be not greater than  $\pm$  0·0001 in., and generally will be within half of this amount. Hot-barrel coated parts will most likely give a greater variation in batch mean coating thickness.

In considering the variation in coating thickness between individual parts of one batch, a statistically normal distribution curve of thickness values was not obtained for either hot-dipped or hot-barrel tinned components. It is interesting to compare this finding with the situation in barrel electroplating of small parts, in which a normal distribution is said to be obtained. 13.14 However, using the treatments recommended, it is possible to keep the spread in coating thickness within one batch of hot-tinned parts to reasonable limits; for example, not less than three-quarters of the batch within  $\pm 0.00005$  in. or  $\pm 25\%$  of the mean coating thickness. What is perhaps more important is that those parts falling on the low side of the range rarely fall below about 0.00015 in. thickness, thus ensuring an adequate quality of finish provided that preparation of the parts and coating procedure have been carried out correctly. In the batch of fifty hot-dipped and centrifuged screws examined, the minimum individual coating thickness was 0.00017 in, when the mean thickness for the load was 0.00026 in.

Geissman and Carlson, <sup>13</sup> working on coating distribution in barrel electroplating, suggest that to obtain only 5% of the batch having coatings less than  $0\cdot00017$  in. thick the average load thickness to be aimed at would have to be  $0\cdot00029$  in., compared with the  $0\cdot00026$  in. for the present hot-dipped specimens. This difference in mean thickness is barely significant, but whereas the batch of hot-tinned screws have 5% with  $0\cdot00017$  in. thickness of coating and none with thinner coatings, in the electroplated parts the 5% of the load would carry coatings from  $0\cdot00017$  in. thickness down to virtually zero thickness. This could mean, for example, that in production a few of these components are certain to have poor solderability and thus cause difficulties on an assembly line. The detrimental effect of inadequate coating thickness, may be greatly magnified by storage





Fig. 4.—Darkening of tin-lead alloy hot-dipped coating (right) after exposure to acetic acid vapour, while the pure tin coating remains unaffected (left).

of the work under poor conditions before it is required to be soldered.

The use of tin or tin-lead coatings for protection against corrosion has been mentioned, and some of the laboratory-processed screws, taken from batches coated by various methods, were examined for porosity by exposure both to a sulphur dioxide atmosphere 15 and to condensing moisture from the atmosphere. It was noticeable that the hot-dipped pure tin coating of 0.00035 in. thickness was superior to all the others. For a thickness of about 0.00025 in, a hot-barrelled coating of 60% tin-40% lead alloy appears to have given slightly better protection to the threaded portion than did the other treatments giving a comparable coating thickness. The 0.0003 in. thickness of tin applied by barrel electroplating gave slightly enhanced protection to the head of the screw (except in the slot) but was considerably inferior at the base of the threads. This is undoubtedly due to a current concentration occurring at the sharp edges, giving an extra thickness of coating at these places, while the depressions in the screw thread were depleted of current and thus received a very thin

Some soldering tests were made with a soldering iron, using pure resin-alcohol flux and 60% tin-40% lead solder, on the screws exposed to humidity. The solderability of the electroplated screws was then much inferior to all the others, and the hot-dipped coatings of pure tin tended to be easier to wet with the molten solder than did the tin-lead alloy coatings. This may be expected, since lead more readily forms a film of corrosion product in the presence of moisture.

Exposure of hot-dipped screws to acetic acid vapour, or vapours of moist oak shavings, caused an even greater difference between pure tin and tin-lead coatings, the latter becoming dark grey in colour (Fig. 4) and then being very difficult to solder.

With regard to the mechanics of coating by the hotdipping or hot-barrel procedures, the latter process appears to be only suitable for small, angular parts which will tumble freely and which have no deep recesses or apertures. Thus, thin-gauge light parts should be avoided, since the presence of the molten coating metal may cause them to agglomerate in a ball or stick to the



Fig. 5.—The Brown-Boveri galvanising machine for small parts utilising a centrifuging head built over the spelter bath.

sides of the barrel and result in bad distribution of the coating. It is thought that the molten coating is transferred from part to part in the main by rubbing together of the surfaces. The inside surfaces of recesses or tubes, such as on tags of Types 1 and 2, therefore rely solely on normal wetting phenomena and on adequate flow of the flux into these parts to attain a coating. It is advisable, therefore, if the tubes are more than about 2 mm. in length, to add an excess of coating metal to the barrel.

Hot-barrel tinning is rather slow, taking approximately 20 minutes for a 20-lb. load but, of course, one operator could easily operate several barrels and thus achieve a production comparable with that obtained by a hot-dipping procedure. The judgment of the "end-point" in barrel tinning requires skill, and an error of perhaps a half-minute either way could result in poorly covered, or at the other extreme, dull and oxidised work. How-ever, the capital outlay on a hot-barrel plant would be very much less than for a hot-dipping unit, chiefly since it is not necessary to purchase a large bulk of tin for the dipping bath.

A hot-dipping procedure appears to be universally applicable to all types of small part similar to those used in this investigation, provided that the correct centrifuging and separating treatments are determined, by prior experiment if necessary. Large loads of work, contained in suitable mesh baskets, may be dipped centrifuged and separated in a period of perhaps one minute, and thus ensure a high production rate. A centrifuge with a heated chamber may be necessary for certain types of light gauge work. For heavier parts which are not completely flat or do not have re-entrant corners or very small apertures, it would seem that

centrifuging is not essential and that a simple separation at high speed, probably at not less than 1,000 r.p.m., may be all that is required. The variation in coating thickness between individual parts coated in this manner is likely to be greater, however, than when centrifuging is carried out.

A certain degree of mechanisation is possible with hotdipping which adds to the attractiveness of this process. Several plants in the U.K. and abroad have achieved excellent results in this manner, but few details have been published in the technical press. One design. thought to be rather commonly employed, follows that of the galvanising machine for small parts, built by Brown-Boyeri in Switzerland and shown in Fig. 5. The parts are carried in a mesh basket which is attached to a vertical spindle above the dipping bath by a suitable quick-release device. The spindle and the basket are lowered into the bath and on withdrawal, a clutch on the spindle engages, when the basket is just clear of the molten metal, and rotates the spindle at a desired speed. thus centrifuging off the excess coating metal. At the end of the desired period the mesh basket is removed from the spindle and the work is passed through a separator. It is not difficult to envisage further developments in which the work is automatically transferred to a separator, quenched washed and dried ready for inspection, without manual handling at any stage. It is likely, however, that mechanical maintenance of such plant would constitute a problem.

One of the greatest practical problems in hot-tinning is the prevention of sticking together of the tinned parts. The type of separator used in the present investigation. when rotating at 1,350 r.p.m., was capable of giving complete separation even of small, completely flat tags (Type 3), provided that they had first been centrifuged. Soft brass parts were found to sustain some damage when separated at this speed, and it is suggested that the blades should therefore be covered with suitable plastic or hard rubber to prevent this damage. Alternatively, the blades could be made of solid plastic, for example rigid polythene, or, as in some presently existing machines, of leather. Another modification considered to be worthwhile is to offset the feed hopper from the top-centre to the down-going side; this should eliminate the tendency for an occasional tinned part to rebound off the impeller blades and out of the hopper, as experienced with the present design of machine. Also, if the blades are in the form of a normal paddle-wheel with adequate clearance on all sides of the blades, there should be

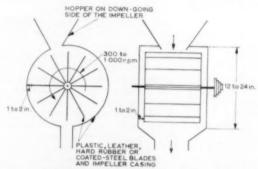


Fig. 6.—Suggested improved design of paddle-wheel type separator giving typical range of dimensions for most types of work.

no possibility of jamming the rotor, as occurred with the present impeller when small parts lodged between the casing and the circular end-plates. These points are shown in the diagram Fig. 6.

There are other types of rotating or vibrating separators in use, but the design used in the laboratory was considered to give a generally quite satisfactory performance.

#### Summary

Examination of small parts such as screws, washers and electrical and radio tags, tinned both in the laboratory and commercially, has shown that a hot-dipping and centrifuging method is more universally applicable than a hot-barrel coating procedure. An acceptable standard of finish was coincident with a mean coating thickness of about 0.0003 in. The variation in coating thickness between individual parts is likely to be such that about 75% of a batch of mean coating thickness 0.0003 in. have thicknesses between 0.00025 and 0.00035 in. This proportion may, in certain instances, alter randomly between approximately 40% and 100% and this phenomenon is not related to any of the known variables. This amount of scatter is not greatly different from that obtained in barrel electroplating, but it is certain that hot-tinning ensures that no individual part has a coating thinner than about 0.00015 in. gives a safe margin where solderability is the criterion. An impeller-type separator rotating at between 575 and 1,350 r.p.m. is able to provide complete separation of parts, which have been coated by a correct procedure, prior to quenching.

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#### Coated Titanium Anodes Agreement

IMPERIAL CHEMICAL INDUSTRIES, LTD., Magneto-Chemie N.V., Schiedam, Holland, and The Amalgamated Curacao Patents Co. of Curacao have agreed to pool their appropriate patents and collaborate to develop and extend the use of titanium anodes coated with an extremely thin film of platinum or similar metal. For some years these firms have been separately engaged in developing coated titanium anodes and independently found that potentially they offered very considerable advantages over conventional materials in industrial electrolytic cells for chemical manufacture, electroplating plant and equipment for converting brine to drinking water.

The greatest field for these anodes is undoubtedly the production of chlorine by brine electrolysis and numerous large-scale chlorine producers are evaluating them for this application, including the general chemicals division of I.C.I., who have been experimenting with cells of this type for more than a year. The anodes are also being used for the cathodic protection of ships and structures such as jetties against sea water corrosion. The metals division of I.C.I. are marketing platinised titanium anodes which are being manufactured in Great Britain by their subsidiary, Marston Excelsior, Ltd., of Wolverhampton.

#### Improvement of Zinc Rich Paints

The Zinc Pigment Development Association is glad to announce that the three of its members who manufacture zinc dust pigments have been awarded a contract by the Expanded Research Program of the American Zinc Institute and Lead Industries Association to carry out a joint programme of research to improve the characteristics of zinc-rich paints. Most of the practical work will be carried out in the Research Laboratories of the Imperial Smelting Corporation, Ltd., at Avonmouth. with the assistance of Amalgamated Oxides (1939), Ltd., of Dartford, and Durham Chemicals, Ltd., of Birtley. A committee representing all three companies and the Zinc Development Association will meet frequently to guide the research.

Initially attention will be given to developing better paints for the priming of motor-car underbodies. Zincrich paints are already finding extensive use for this purpose in the U.S.A., where the vast quantities of salt used each year to remove snow in the northern cities have created a serious corrosion problem with chassisless construction. With the co-operation of the motor industry it is hoped to be able to formulate paints with better adhesion, flexibility and welding characteristics than those currently available. Later on, work will be done on paints for structural steelwork and marine applications.

The Expanded Research Program was established in 1958 to sponsor fundamental and applied research on zinc and lead with a view to developing the uses of the metals and their derivatives. The Program is supported by metal producers and mining companies in the U.S.A. and the British Commonwealth, and is being carried out in several countries. The zinc and lead industries will make the results available for the benefit of the whole of the industries concerned.

#### First Nickel from Thompson

SHIPPED at the Hudson Bay port of Churchill, at the beginning of the very brief open season, the first consignment of nickel from Thompson, Manitoba, arrived at Swansea on 12th August, en route for the Clydach works of The International Nickel Co. (Mond), Ltd. There, the 2,700 tons of full-sized cathodes will be cut to 4 in. squares and packed for distribution to European steel mills and other consumers. Inco's \$185 million Thompson project is expected to increase the world's annual nickel supply by some 37,500 tons, half of which is destined for consumption on this side of the Atlantic, to meet the steadily increasing demand. Formally opened as recently as the end of March, the new plant is expected to provide Europe with 6,000 tons before ice closes the direct shipping route from Churchill this year.

# Steel Castings Research

## B.S.C.R.A. Reports Progress

THE second year of the British Steel Castings Research Association's second quinquenium of D.S.I.R. grant has been characterised by consolidation following the extensions to the experimental foundry and laboratories, which were carried out during the previous year. The installation of new plant and equipment, notably of a 56 lb. vacuum induction melting furnace, together with a small addition to both the research and administrative staff, have produced the optimum scale of effort from the available resources.

#### Special Assistance to Industry

The Association's external activities have shown a marked increase, which is partly attributable to the Special Assistance to Industry Scheme, initiated in October, 1959, whereby a grant on especially favourable terms has been made available by D.S.I.R. to support a three-year programme of activity designed to secure greater application in industry of the results of research. Among subjects which have received attention since the scheme was introduced are the removal of carbon from molten steel by oxygen blowing; the performance of commercial heat treatment furnaces; and work simplification schemes, particularly in connection with core shop and fettling shop layouts.

During the next year it is intended to continue the studies of the performance of commercial heat treatment furnaces: apart from specific advice to firms leading to improvements in heat treatment technique, the information accumulated will provide a valuable basis for discussion with manufacturers on possibilities of improving the design of heat treatment furnaces. Because of the time required to complete a work simplification study, it is proposed to encourage member firms to use their own staff in such studies, leaving the Association's specialist to provide an advisory service in planning exercises and interpreting results. Two new projects to be included in the scheme are the ultrasonic examination of steel castings for defects, and the preparation and application of mould paints: demonstrations will be given in individual foundries or appropriate centres.

#### RESEARCH PROGRESS

The progress of research during the year 1960-61 is summarised in the Association's Eighth Annual Report, from which the following information is extracted. In addition to the aspects covered here, work is also in progress on industrial health, particularly in relation to dust in the atmosphere and noise levels in the foundry.

#### Steelmaking

Desulphurisation of Liquid Steel

The experimental work on the reversed slag process, working on both magnesite and dolomite hearths, has been completed, and the following conclusions have been drawn:

(a) The rate of sulphur removal can be increased.

especially at low sulphur levels, by agitating the bath by the injection of nitrogen well below the slag/metal surface and/or by dispensing suitable powdered materials in the nitrogen stream.

(b) When working on a magnesite hearth, it is possible to produce steel with a low sulphur and a low hydrogen content by a reversed, two slag process—that is a reducing period, followed by an oxidising period. With a dolomite hearth, this has not been possible owing to sulphur reversion from the hearth to the metal during oxidation.

Works trials have also been carried out in a 5 ton are furnace with a dolomite hearth. As a result of these trials it has been found that the injection of calcium carbide alone was not effective in promoting rapid desulphurisation and that to do this required more complex powder mixes, such as calcium carbide/magnesium/fluorspar or burnt lime/magnesium/fluorspar. When using such mixes, rapid desulphurisation to low sulphur levels has been obtained.

As in the experimental work, it has not been possible in these trials to obtain very low sulphur levels by the application of the reversed two slag procedure because of sulphur reversion from the hearth. It has been possible, however, to obtain steel of a low hydrogen content (approx. 2 ml./100 g.) with sulphur contents of less than 0.02% by such a reversed two slag procedure.

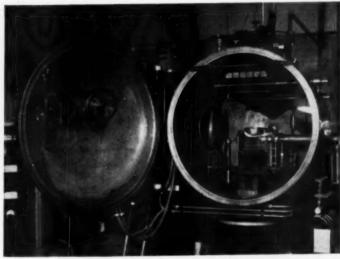
#### Pinhole Porosity

The severity of pinholing is now measured by a radiographic technique and, using this method, the steelmaking variables are being investigated. It has been found that additions of strong deoxidisers, such as aluminium, titanium and zirconium, drastically reduce the severity of pinholing, and that zirconium is the most effective in this respect. So far no relationship has been found between the severity of pinholing and atmospheric humidity. The effects of bismuth and selenium additions have also been studied, but neither of them has proved effective in eliminating pinholes.

Test castings have been made in carbon-free iron, and the number of pinholes was thereby markedly reduced from the usual number of about 100–300 obtained with steel made to B.S. 1617, Grade A, down to about 10 or 20. These experiments, together with the work on deoxidants, offer further substance to the belief that the carbon-oxygen reaction is of prime importance in pinholing.

Microscopic and radiographic examination of mould pour-out eastings showed that the growth of the pinholes is preceded by the formation of fine capillary tubes at the mould/metal interface which apparently act as nuclei for the pinholes.

Methods for artificially increasing the hydrogen and nitrogen content of the steel have been developed. Work is proceeding to study the effect of these on the severity of pinholing, both in the presence and absence of carbon. A start has also been made on the study of



The G.E.C.-V.I.A. vacuum induction melting furnace installed in the Association's experimental foundry during the year under review, has a 36 in. diameter horizontally mounted chamber, and is capable of melting 56 lb. of steel.



#### Moulding and Casting

Factors Affecting the Surface Quality of Steel Castings

Mould Paints and Washes.—Further attention has been paid to the formulation of water-based zircon mould washes. In a number of member foundries the recommended alginate suspended washes are being used very satisfactorily while in others some modifications have been made to suit individual needs. One difficulty encountered during the foundry trials has been the limited bench life of the alginate paint mix, and while this does not detract from its usefulness, when prepared in the foundry using suitable mixing equipment, it becomes a serious disadvantage when the paint is prepared commercially.

Work has shown that in order to prevent metal penetration under high ferrostatic heads, a comparatively thick coating of paint must be applied on to the sand surface. Since this involves at least three successive applications of paint, which is a time consuming operation, methods of obtaining the thickest possible coat in one application are being studied.

The possibility of using self-healing paints, i.e. based on refractories that partially melt at molten steel temperatures, has received some attention. Some selected combinations of SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub>, and SiO<sub>2</sub> and MgO have shown promise, but difficulties are foreseen in applying such paints in practice.

Further work on inflammable type paints has resulted in the formulation of suitable paint compositions that are being used successfully in member foundries.

Metal Penetration.—The pressure casting technique developed for simulating high ferrostatic pressures has been employed for determining the resistance to metal penetration offered by various types of sand mixes and



This hydraulically operated sand testing machine of 5 tons capacity was designed and built by the Association's staff. It is used for the compression testing of sands, such as certain CO<sub>2</sub>/sodium silicate or organically bonded mixtures, that are too strong for testing with normal sand testing equipment. Provision is made for the fitting of a furnace in order to determine high temperature properties of sand mixtures.

for studying the mechanism of penetration.

Among the factors studied has been the effect on metal penetration of ramming density, the grain size of the sand, the results of adding various proportions of fine fillers, and the use of zircon sand. A most important conclusion reached so far is that complete resistance to high ferrostatic pressures is obtained only from sand mixes of silica or zircon, having a grading approaching the fineness of the refractory base used in paints. The problem now is to decide which is the more practicable and economical procedure, viz., the application of a thick coating of paint on to a normal silica sand or the use of these very fine sand mixes, some of which, especially the siliceous ones, may have poor thermal stability and be more liable to expansion defects.

By a thermal analysis of the sand during and in the absence of metal penetration, some indication has been gained of the speed of metal penetration and how metal penetration affects the thermal conditions in the sand.

Factors Affecting the Erosion of Moulds by Molten Steel.—A casting technique has been developed by which the amount of sand erosion taking place during the passage of molten steel can be measured quantitatively. Using this technique, the effect of changes in bulk density of the sand, the type of bonding agent, and the sand grain size have been investigated, and also the effect of varying the amount of metal poured. It has

been found, contrary to published opinion, that erosion increases progressively with the amount of metal

noured

The amount of erosion is related to the density of the sand compact and to the grain size of the sand employed. Erosion is minimised by harder ramming and by the use of a finer sand, but in both cases there is an increased tendency towards expansion scabbing defects. Varying the type of bonding agent does not appear to affect the amount of erosion, but does affect the density at which expansion defects begin to occur. By increasing the resistance of the sand to expansion scabbing, cereal additions allow harder ramming and thus minimise erosion.

Thermally stable sands, e.g. zireon of very fine grain size, greatly reduce the amount of erosion without producing expansion defects, but equally thermally stable sands, e.g. molochite and olivine in coarser gradings, erode similarly to silica sands. This suggests that erosion is not a function of thermal instability, but is due to the ability of the metal to penetrate the surface of the sand

and force out the sand grains.

The Friability of Green Sand Moulding Mixes on Air Drying.—A correlation has been found between the friability of green sand compacts on air drying and the dry compression strength of the sands. The correlation is not affected by variation in sand grain size or by the type of bonding agent, the only exceptions being sand containing additions, such as dextrin, that harden the surface on air drying. The addition of wetting agents and moisture retaining materials to the sand have little effect on friability, and the greatest benefits are obtained by the use of hardening sprays.

So far, little correlation has been found between friability of the sand and its resistance to erosion by molten metal. The prevention of friability seems to be more advantageous, therefore, in lessening the chance of loose particles of sand falling into the mould during

movement and handling.

#### CO<sub>2</sub>/Sodium Silicate Process

A simple test casting and testing procedure has been developed for determining the knock-out properties of  $\mathrm{CO}_2$  process core sand mixes. Several types of sodium silicate and a wide range of organic and inorganic

additives have been investigated.

Several organic materials, in additions of 1.5% by weight, have been shown to be fairly effective in assisting core removal after casting without seriously impairing core strength properties. Among the more beneficial additives tested were pelleted pitch, dextrin and coal dust, and certain proprietary organic additives.

Certain inorganic materials were also effective, but higher additions were required than in the case of the organic materials. Refractory clays, such as ball clay or china clay, were the most promising of this group of materials. Calcium carbonate, either pure or in the form of ground chalk, was found to be moderately effective, whereas sulphate materials, having different decomposition temperatures, were less beneficial.

Ammonium chloride in the form of pellets gave fairly good results without lowering core quality, but rough castings resulted if the pellets were present at the metal/core interface. In granulated form, this breakdown agent gave very good results with less pronounced

surface roughness.

Some improvement in core collapsibility was obtained by using silicates of higher silica: soda ratio, but very high ratios (3:1) gave poor quality cores. The importance of keeping the silicate addition as low as possible has also been demonstrated.

#### Moulding Sand Compaction

Work at Sheffield University.-This work is now complete and has been concerned primarily with the flow properties of bonding clays in relation to their effect on the flowability of moulding sand mixtures. Three methods have been used to study the flow properties of clays in the range of clay/water ratios found in moulding sands, viz. a capillary flow viscometer, a shear plate viscometer at constant stress, and a parallel plate viscometer at constant straining rate. As the conditions of flow in the capillary viscometer differed from those to which the clay films are subjected during the compaction of moulding sands, the method was discarded. Stress/strain curves at constant stress and at constant strain rates have been determined for a number of clays and the effect of film thickness and the clay/ water ratio studied. The results are being analysed in relation to their practical significance in terms of the compaction characteristics of moulding sands.

Work at B.S.C.R.A. Laboratories.—The study of the effects of grain size and grain size distribution on the packing density of the sand has been extended to include moist bentonite bonded mixtures. Compared with the dry, unbonded mixes, these additions caused a marked reduction in density, although showing the same trends as regards bulk density. There was no correlation between bulk density and permeability and thus, when the degree of compaction is increased in this way, casting defects, such as metal penetration, are not necessarily reduced.

#### Mould and Core Bonding Agents

Apparatus has been constructed for determining the amount of sagging of organically bonded sands during baking, and the resistance to sagging of several airsetting binders has been measured. The effects of the amount of binder, clay additives and air-set strength have also been investigated.

The possibility of hastening the setting of air-hardening binders by "gassing" with air or oxygen is under investigation and a series of special binders (styrenated alkyds) that harden by evaporation of a solvent are

being tested.

The properties of cereal binders are being evaluated and the aspects covered include the effects of cereal additions on the strength properties of both clay and oil bonded sand mixtures, the bonding properties of the cereals alone, and the effect on mould properties as indicated by wet-tensile, thermal shock, friability and erosion tests.

#### Data Sheets on Moulding Sands

Work is continuing on the determination of the properties of further sands for inclusion in a revised edition of the special publication "Data Sheets on Moulding Sands." A simple apparatus for measuring the specific surface of sands has been constructed and standardised for use as a routine test.

#### Exogenous Inclusions in Steel Castings

Instead of activating silica sand grains coated with ceria, it has now been found possible to activate the ceria alone and then use it to coat and label silica sand grains, this being much more economical and just as effective as the earlier procedure. Tests have shown that the activated coating is firmly affixed to the sand grains, even after being in contact with molten steel for some time. Activated sand compacts have been placed in the runner system and in the mould cavity and loose grains of sand have been placed on the bottom of the mould. Only when activated sand was used in the runner system were active sand grains removed to another part of the casting. These inclusions always took the form of large agglomerates of sand grains in a slaggy matrix and were situated at or just below the surface of the casting. Even when the activated sand was added to the metal stream during pouring, the sand

grains still coalesced to form larger inclusions at the surface of the casting. Chemical analyses of the inclusions showed them to be mainly of SiO<sub>2</sub>, but containing a high percentage (10%) of MnO.

#### Factors Affecting the Soundness of Steel Castings

A critical review has been made of the available literature relating to the soundness of steel castings and a bibliography of over 300 references has been prepared.

Work has commenced in investigating the foundry variables that affect feeding, and work so far has indicated that the degree of mould compaction and pouring temperature within practical limits have little effect on soundness.

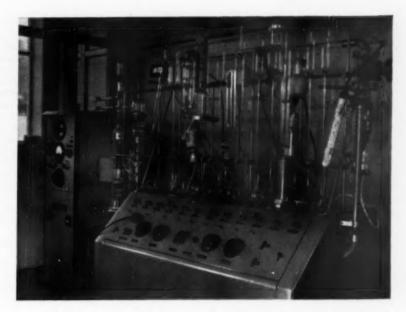
A detailed study of the literature on the liquid and liquid to solid contraction of steels has disclosed very little information relating to alloy steels. Methods are, therefore, being developed for determining the shrinkage and feeding characteristics of such steels.

#### Metallurgy

#### Microporosity in Steel Castings

In a preliminary series of experiments, it was shown that microporosity varied with the width of the freezing range, as controlled by the carbon content. A further series of steels was, therefore, made within closer compositional limits and covering a wider range of carbon content. For each carbon content, a series of pouring temperatures was used representing differing degrees of superheat above the liquidus temperature. The castings were assessed for microporosity content by the point counting of semi-microradiographs.

The results indicated that the degree of superheat had no significant effect on the incidence of microporosity. Further, steels with carbon content up to 0.45% contained no more microporosity than 0.10% C



A Wild Barfield vacuum fusion apparatus for the determination of oxygen in steel forms part of the equipment of the gases-in-steel laboratory.

steels. In steels containing 0·45–0·85% C, microporosity appeared to increase with increasing carbon content. It was therefore concluded that in the commonly used steels with carbon content lower than 0·45%, superheat and freezing range do not, to any practical extent, influence microporosity.

#### Heat Treatment of Steel Castings

Laboratory work on the effect of heat treatment on mechanical properties of cast steels has continued. It has been shown that, in a 1½% Cr-Mo steel, an anneal prior to hardening and tempering is not particularly beneficial to mechanical properties. Work is continuing on the effect of hardening times for this class of steel.

#### Intergranular Fracture in Steel Castings

This investigation, which is now nearing completion, is being conducted in the Metallurgy Department of Sheffield University under the direction of Professor A. G. Quarrell. It has been shown that steel castings containing precipitates of aluminium nitride at the prior austenitic grain boundaries will fracture in an intergranular manner. Variations in the carbon and sulphur contents of the steel have an important effect on the incidence of this intergranular fracture. The current opinion following this work is that for a given amount of aluminium nitride at the grain boundaries, the factor that determines whether an intergranular or a ductile fracture will occur is the relative weakening powers of the nitrides at the grain boundaries and of the sulphur particles and other inclusions within the grains. If the grains themselves are strengthened, as by the addition of carbon or other alloying elements, then an intergranular fracture will occur more readily. Work is continuing to test this hypothesis in all its aspects.

#### Microsegregation in Cast Steels

Pure iron binary and ternary alloys have been used for *ab initio* work on microseg regation. Several methods have been applied in an attempt to obtain quantitative measurement of microsegregation. All of these, including the scanning electron probe microanalyser, have so far proved unsuccessful on the special alloys made, and also on conventional and special cast steels.

Work under this heading is also being done under contract from the War Office on the development of high tensile cast steels. The results obtained so far have not been very encouraging, as the composition on which most of the work has been done appears to be difficult to cast completely sound, and at the 100 tons/sq. in. tensile level the ductility is lower than is desirable. The work is continuing with an investigation of the effect of solidification rate on the mechanical properties obtained at high tensile levels.

#### Factors Affecting the Properties of Cast 13% Cr Steels

Initially, the effect of steelmaking variables, including deoxidation practice, are being investigated. Deoxidising additions have included aluminium, selenium, titanium and zirconium, and heats have been made using 100% and 50% of 13% Cr process scrap and none at all in the furnace charge. Mechanical testing and micro-examination of these steels is in progress.

Mass, in so far as it affects solidification and cooling rates, will later be studied. Also, the vacuum induction melting furnace will be used to investigate the effect of gas content and residual elements.

#### Deoxidation of Cast Steel

The recent installation of the 56 lb. vacuum induction melting furnace will make possible a fundamental approach to the problem of deoxidation, its effect on non-metallic inclusions and properties of cast steels. Initially, the separate and combined effects of aluminium, oxygen and sulphur on the type of inclusions in iron-carbon alloys will be determined. This will then be followed by a study of the effect of other deoxidisers and desulphurisers, with and without aluminium (e.g. Si, Mn, Ca, Ti, Zr, Ce).

Work carried out on the vacuum furnace so far has been concerned with finding suitable materials for containing the melt and suitable charge materials, determining the losses and control of composition during melting, and developing an operating technique for the plant as a whole.

#### Plant Engineering

#### Sand Reclamation

Work has continued in several fields of investigation, including methods of sand scrubbing, the treatment of effluent water and the estimation of the total clay content. Two pilot units have been designed and constructed in the Association's workshop for reclamation of moulding sand by an intensive dry scrubbing action. One unit is a dry pneumatic scrubber. Up to the present it has not been possible to reduce the clay content below 2.8% with this apparatus. The second unit is essentially a wire helix rotating in a column of sand to produce an effect similar to that of an Archimedean screw. Preliminary tests on this type of apparatus are promising and have verified the theoretical deduction

that a cleaner sand may be produced than by other dry reclamation units.

Work on the wet system has been mainly confined to the treatment of effluent water. Trials at the Association have shown that, by the addition of chemical reagents to create flocculation and permit fast settling rates, 90% of the effluent water may be recirculated quickly and economically. A summary is being made of the technical and economic aspects of wet reclamation methods.

Work has continued on the assessment of cleaned sands. The amount of live clay in a sand mix can be accurately estimated by making careful use of the nickel ion adsorption technique. Also, the normal A.F.S. clay test produces more accurate and reliable results with a sand containing live and dead clay if it is previously milled, preferably in water, to prevent the loss of fines. Work is in hand in attempting to relate the clay and corresponding alumina contents in a sand mix.

#### Efficiency of Shot Blasting

Testing has continued on steel shot and on cut wire, using the modified method of test, in which additions are made to the charge at regular intervals to maintain it at its original level.

A study of the effect of air resistance on abrasive velocity has been made and tests carried out at various projection speeds have revealed large variations in abrasive breakdown rates. It is hoped that this work will eventually lead to the determination of critical abrasive impact velocities.

The effect of sand contamination of abrasives on the wear of impeller blades has been carefully studied. It was found that the presence of 2% sand by weight increased blade wear by up to five times, and 5% sand increased blade wear by as much as 15 times. These figures illustrate the need for efficient operation of abrasive separators in commercial shot blasting plant.

The testing of a number of cut wire samples of particle sizes ranging from 0.032 in. to 0.080 in. have shown that, under testing machine conditions, cleaning efficiency is highest when using particles between 0.050 in. and 0.060 in. dia. This finding should also apply in full size blast cleaning equipment.

Work is continuing on wire abrasives to determine the effect of the various physical and metallurgical properties and manufacturing processes on their blast cleaning efficiencies. For this work, a series of wires 0.060 in. diameter is being obtained direct from the wire manufacturers.

#### Atmospheric Pollution from Steelmaking Processes

Data to assist an assessment of the contribution to atmospheric pollution by the foundry steelmaking processes, viz. electric arc furnaces and Tropenas converters, have been obtained by the Association and previously reported. This information has been very useful in discussing the subject with the Alkali Inspectorate and the staff of member companies.

Recently, tests have been made on Tropenas converters fitted with a wet collector for cleaning fume. As expected, the emission from the collector retains the characteristic brown colour, although the Association's tests have confirmed that approximately 75% of the total weight of dust and fume generated by the converters was retained by the collector.

Comparison of the results from sampling fumes emitted from a 4 ton electric arc furnace, using ore boil and oxygen injection, respectively, has shown that the peak emission of fume when ore boiling is much less than when oxygen boiling, but the total weight of fumes per ton of steel melted emitted during the whole melt using ore boil is definitely not less than when using oxygen injection and maybe more.

Samples have been taken also in a member foundry on an experimental installation, in which fumes have been extracted from a 2½ ton electric are furnace through an opening in the side wall of the furnace. This experiment has confirmed that fumes can be controlled using a much lower exhaust rate than with hood extraction.

#### Factors Affecting the Efficiency of Mould Drying

A number of measurements on mould drying rates in various drying stoves have been made by members of the North-East Coast Association of Steel Founders Regional Technical Committee. These studies have supplied valuable information, which has been analysed, and a progress report prepared. The preliminary analysis showed that there is a wide scope for improvements in the thermal efficiency of various stoves, in particular their thermal insulation, and has indicated further lines

of approach to the study of the drying process. An important factor in considering the mould drying process is the "strike back" of moisture and a study of this aspect of the problem has been carried out by the Foundry Processes Section.

#### Removal of Excess Metal from Steel Castings

Of the various methods of metal removal, grinding and flame cutting are being studied in the first instance. A literature survey has been made to assist in establishing a testing procedure to obtain data on the relationship between wheel wear in grinding machines and the peripheral speed and the pressure applied to the wheel face. There is sufficient evidence to suggest that considerable saving can result if the peripheral speed of a grinding wheel is kept constant. As a result of the survey of available equipment that can be incorporated in a grinding machine to give a constant peripheral speed to a grinding wheel during its life, a suitable equipment has been offered to the Association and it is hoped that tests will be initiated in the near future. In conjunction with the suppliers of gases and equipment, tests have been initiated in the Association's laboratories to compare the relative merits of oxyacetylene and oxy-propane cutting.

#### S.C.O.W. Tinplate for U.S.A.

THE Steel Company of Wales is to start supplying regular shipments of tinplate to the United States of America. During October and November 11,200 tons of tinplate will be shipped into San Francisco and other ports of the West Coast for delivery to the Continental Can Company and the American Can Company. To conform with the current trend in the United States, the bulk of these orders will be delivered in coil form and will constitute the biggest output of coiled tinplate hitherto produced by the company.

The Hon. M. J. Layton, sales controller of The Steel Company of Wales, commenting recently on these orders, said: "To enable us to maintain the best possible service to all our customers, it is essential that we keep in close touch with the qualities and practices required in the American tinplate market. The best way of doing this is to be suppliers to U.S. can manufacturers. In accordance with the practices developed in recent years in America of using tinplate in coils, the bulk of the orders will be supplied in this form. We have been able to accept these orders because of our decision taken some years ago, to install equipment for the production of coiled tinplate."

#### **Dorman Long Development**

The first major orders in connection with the present stage of the Dorman Long (Steel), Ltd., development programme have now been placed. Davy-United, Sheffield, have secured the order for a 48 in. × 130 in. high-lift slabbing and blooming mill at Lackenby, at a price of £1,300,000. Intended to roll 25,000 tons of ingots per week, to produce blooms and slabs, the mill will be sited alongside Dorman Long's existing universal beam mill at Lackenby. The slabs produced will supply the existing wide plate mill at the company's Redcar Works, and the blooms necessary to meet the increasing requirements of the company's medium and light

finishing mills. The mill will also supply in due course a new universal 4-high plate mill capable of producing 60 in. wide plates with rolled edges. The soaking pits, valued at approximately £700,000, will be supplied by Priest Furnaces. Middlesbrough.

The £450,000 order for the blooming and slabbing mill electric drive has been placed with Associated Electrical Industries, Ltd. This new drive consists of two 5,000 h.p. motors driving top and bottom mill rolls independently. The motors are supplied from grid controlled reversing rectifiers. Orders will shortly be placed for the mill auxiliary motors, control gear switchgear and electrical distribution equipment.

The Wellman Smith Owen Engineering Corporation, Ltd., will supply ingot stripping and soaking pit cranes to the value of approximately £400,000. All the foundation work for the mill will be carried out by the bridge department of Dorman Long, who did the foundations of the Lackenby steelplant and universal beam mill. The mill buildings will be supplied and erected by Dorman Long (Bridge & Engineering), Ltd., Redpath Brown & Co., Ltd., and the Tees Side Bridge & Engineering Works, Ltd.

The lining of G.R. Supermag magnesite bricks contributed largely to the achievement of a record throughput of 530,304 tons of hot metal recently obtained from a 1,200 tons capacity hot metal mixer at the Abbey Works of the Steel Company of Wales. This is a record for British steelmaking and the first time that the half million ton mark has been reached in this country.

Schloemann a.g., Düsseldorf, are now supplying their forging presses with a digital control for automatic forging and planishing, which enables forgings to be produced uniformly, to a high degree of dimensional accuracy. As the use of calipers for gauging is dispensed with and the production cycle proceeds without interruption, a high output is obtained.

# Brittle Fracture

# Descriptive Bibliography-Mainly Post-War

By E. C. Pigott, F.I.M.

On this important subject, the bibliographer gives full references to 135 notable contributions. A brief description of each embodies the key points, so allowing of trustworthy overall perspective. A matter of much interest is the evolution of currently accepted concepts, which are reflected in steelworks and fabricating practice, in many recent standards on structural steel, and in the revised rules of inspection societies, themselves responsible for part of the spectacular success won in the campaign waged against catastrophic brittle fracture.

Acker, H. G. Welding Research Council (New York), 1954, Bulletin Series No. 19.

A critical review of ship failures; 6,000 vessels affected in the period 1938–1954. The main factors were welding notches, design notches and cold notch-sensitivity. The problem of hatch corners and square cut-outs was solved, but bilge keels, connections of tanker longitudinals to bulkheads, etc., call for improvement. Notch-toughness of hull plate is the main line of defence.

Anon. Admiralty Advisory Committee on Structural Steel (London), 1956, Report No. P1 (H.M.S.O. 20—165—1).

A review of brittle fracture in the U.K. Improved welding technique and a notch-tough acceptance test are required.

Akita, Y. Welding J. (New York), 1953, 32, 475.

Thicker sections have higher transition temperatures.

Almar-Naess, A. British Welding J., 1957, 4, 88-102.

Bend tests. The internal energy released by the advancing crack maintains propagation. Transition temperature taken as that at which half the fracture is crystalline. In service, static stress distribution and stiffness are influential.

Andige, A. Assoen. Tech. Maritime et Aeronautique, 1953.

Damage in Liberty ship *Grenoble* in calm weather while at jetty. Thermal stress in plating was caused by heating of oil-fuel tanks.

Anon. Ship Structure Committee (Washington), 1955, Report No. SSC—87.

Stored elastic energy is converted into surface-tension energy.

Anon. "Metals Handbook," Amer. Soc. Metals (Cleveland, Ohio), 1954 Supplement, 2.

Technical summary.

Anon. Technical Report, British Engine Boiler and Electrical Insurance Company (Manchester), 1954, 2.

Brittle fracture in swing bolts was due to a surface layer of severely strain-aged material.

Anon. Shipbuilding Research Assocn. of Japan, Third Research Committee, Report No. 2, October, 1954.

Deoxidation and semi-deoxidation depress the transition temperature; the heat-affected welding zone is markedly brittle; riveting increases the absorption of energy. Anon. Shipbuilding Research Assocn. of Japan, Report No. 14,

Schnadt-Lebigh-bend, Charpy impact, Kahn tear, Tipper and Vanderveen tests reveal the transition temperature. All these tests are reliable, provided the notch is sharp.

Anon. Ship Structure Committee (Washington), 1955, Report No. NBS-5.

All 100 ship fractures examined started at a design or fabrication defect.

Anon. German Symposium. Tecknic (Berlin), 1954, 9, 573.

Anon. Revue Soudure (Brussels), 1959, 3, 147-152.

Resistance to brittle breakage of cracked steel plates.

Baldwin, W. M., and Evans, E. B. Ship Structure Committee (Washington), 1953 Report, No. 88C—64.

Confirmation that quench-ageing is responsible for much of the brittleness of the zone outside the weld area.

Barr, W., and Honeyman, A. J. K. J. Iron Steel Inst., 1947, 157, 239.

High Mn/C ratio gives low transition temperatures.

Barrett, C. S., and Mahin, W. E. Ship Structure Committee (Washington), 1954, Report No. SSC-70.

Most failures occurred at welds; crack-arresting plate is advisable. Few failures occur at heat-affected zones.

Barrett, C. S. Trans. Amer. Soc. Metals (Cleveland, Ohio), 1957, 49, 53.

Metallurgy at low temperatures.

Barenblatt, G. I. Doklady Akademii Nauk, S.S.S.R., 1959, 127, 47-50.

Under load, micro-cracks grow to visible dimensions.

Beachem, C. D. Naval Research Laboratory (Washington), 1959, Report 5263; (P.B.151352).

High-strength sheet steels; fracture mechanism.

Biggs, W. D. The Welder (Murex, Waltham Cross), 1956, 25, 22, 48, 72; 1957, 26, 2.

The Tipper test is preferred. Robertson designing is uneconomic. Slag, lack of fusion and undercut are the worst defects. Fully-killed steel is required. Fine grain obtained by dint of a low finishing temperature is unstable to welding heat.

Biggs, W. D. "The Brittle Fracture of Steel," 1960, London, Macdonald & Evans, Ltd., pp. 420.

Comprehensive text book for metallurgists and engineers.

- Boodberg, A., et al. Welding J. (New York), 1948, 27, 1868.

  Tests on internally notched plates up to 72 in. in width
- Boyd, G. M. Engineering (London), 1953, 175, 65, 100.

Successive disc-shaped cracks ahead of the main fracture front.

Boyd, G. M. Structural Engineer (London), 1955, 33, 11.

Initiation requires greater stress than propagation; at low stress, a "notch" is required; the notch root tends to be brittle; welding can cause strain-ageing.

Boyd, G. M. Ship Structure Committee (Washington), 1959, Special Report SSC—125, 20.

Shear and cleavage fractures; initiation; Charpy test; failures.

- Brown, B. S. Engineering News Record, 1919, B2, 974.
  Failure of molasses tank, Boston, U.S.A.
- Brown, D. P. Welding J. (New York), 1952, 31, 765. Historical sequence of failures.
- Buckner, H. F. American Society Mechanical Engineers, 1957, No. 57—A—189.

Griffith and Irwin-Orowan models.

Capus, J. M. Revue Mét. (Paris), 1959, 56, 181-9.

Lead, arsenic, antimony and tin promote notchbrittleness of Ni-Cr-Mo steels.

Christensen, N., and Augland, B. Brit. Welding J., 1957, 4, 69-87.

Tests on 14 ship-plate steels.

Comm, J. (Ed.). Shipbuilder and Marine Engine Builder (Newcastle upon Tyne), 1954, 1.

Week (Cambridge) considered brittle fracture in rivetted ships; welds in laminated plate have an unconnected joint. Craters formed by repeated striking of arc on the metal surface produced notches; an earthed striking-plate is required.

- Cottrell, C. L. M. Nature (London), 1952, 170, 1,079. Effect of hydrogen; diffusion embrittles.
- Cox, J. J. Ship Structure Committee (Washington), 1954, No. SSC—66.

Formation of twin crystals below -170° C.

Curll, C. H. U.S. Research Report, 1960, 33, 545.

Non-linear correlation between sub-size and standard Charpy.

Dedleu, J., and Delannoy, R. Revue Mét. (Paris), Mem. Sci., 1959, 56, 567-581.

Influence of initial heat treatment on the transition temperature.

Dick, W. Stahl u. Eisen (Düsseldorf), 1960, 80, 1,588.

Tests on 37 open hearth and converter steels (0.04-0.21% carbon) showed deoxidation to be highly beneficial. The Charpy test is a reliable criterion of resistance to fracture.

Drucker, D. C., et al. Ship Structure Committee (Washington), 1954, Serial No. SSC—69.

Propagation studies are more important than initiation studies. In tubes the main factor is longitudinal stress. Elongation relieves stress. Microscopic areas of welding slag may initiate cracks. Low width/thickness ratio favourable. Fast propagation promoted by stored elastic energy; opposed by absorption of energy.

Evans, E. B., et al. Ship Structure Committee (Washington), 1953, Nos. SSC—60 and SSC—61.

The brittleness of the zone adjacent to welds is due to quench-ageing at 650-1.300° F.

Feeby, F., et al. Ship Structure Committee (Washington), 1953. Document FE4/401.

Failure of two Fawley oil tanks. Below the critical temperature, the brittle breaking stress is constant. At 40° F., fracture occurs at a stress of 6-7 tons/sq. in.

Felix, W., and Geiger, T. Sulzer Tech. Review (Winterthur), 1956, 38, 14.

Actual fracture preceded the cracking of individual crystals having slip-planes in line with applied stress, so preventing stress-reduction by flow before the cohesive-strength was exceeded in the cleavage plane.

Flossner, H., and Matthaes, K. Schweizer, Archiv. Wissenochaft. Technic (Solothurn, Switzerland), 1957, 23, 249-258.

Influence of temperature, composition and velocity.

Fox, F. A. Science News, 1955, No. 36, 75-84.

An accurate presentation of brittle fracture of structural steel for the general reader.

Garmo, E. P. de. Welding J. (New York), Res. Supp., 1948, 27, 505.

Full scale tests on ship hatch-corner assemblies.

- Gertsman, S. C. Canad. Dept. Mines Techn. Survey, Phys. Met. Div. Res. Special Proj. 1960, 29.
- Gilman, J. J. Acta. Met. (Toronto), 1960, 8, 665.

Improvement effected by grain-size reduction is limited.

Graaf, J. E. de, and Veen, J. H. van der. J. Iron Steel Inst., 1953. 173. 19.

The notched slow bend test is more reliable than the Charpy test.

Hall, E. O. J. Mechanics and Physics of Solids (London), 1953, 1, 227.

Brittle fracture of steel is not analogous with glass fracture. The plastic energy in single crystals is slight.

Hall, W. J., et al. Welding J. (New York), 1957, 36, 18.

With large steel plates, attention to material, geometry, temperature and stress-level is essential.

Hall, W. T., et al. Ship Structure Committee (Washington), 1958, Report No. 111, 86.

Propagation in wide steel plates, ice cooled, loaded and subjected to impact.

- Hartbower, C. E. Welding J. (New York), 1957, 36, 4018.
  Test variables.
- Hartbower, C. E. Welding J. (New York), 1957, 36, 4948.

Initiation of test cracks by a low-energy blow. The brittle crack grows by continuous release of elastic stress field; ductile crack-growth requires plastic-deformation work.

Hauttmann, H. Schiff u. Hafen, 1957, 507.

Notch-toughness of L.D. (Linz-Donawitz oxygenblown converter) steel.

Hechtman, R. A. Soc. Naval Architects and Marine Engineers (New York), 1953.

All welded ships' failures occurred below 50° F. Notches at sheared and flame-cut edges; accidental arcstrikes; material was under-deoxidised. Cold working should be avoided.

Hodgson, J., and Boyd, G. M. Trans. Inst. Naval Arch. (London), 1958, preprint pp. 25.

Failures, safety standards and notch-toughness tests.

Imbermbo, E. A., and Ginsberg, F. Ship Structure Committee (Washington), 1959, Report No. 180, 21.

The transition temperature of low carbon-high manganese semi-killed ship steel plate, \(\frac{3}{4}\), 1\(\frac{1}{4}\) and 1\(\frac{3}{4}\) in thick, evaluated by Vanderveen notched slow-bend test.

Irwin, G. R. American Society Mechanical Engineers, 1957, No. 57—APM—22.

Stress intensity at the crack-end is proportional to the square root of the cracking force.

Irwin, G. R., et al. Proc. Amer. Soc. Testing Materials, 1958, 58, 640-660.

Strength in relation to initiation and propagation.

Irwin, G. R., et al. Naval Research Laboratory (Washington), Report 1958, 5222 (P.B. 151131).
Griffith theory.

Izdinsky. Zvar. Sbornik (Bratislava), 1959, 7, 180-192.

Notch effect at the edge of welded seams (in river boat hulls) is most influential.

Johnson, H. S., and Stout, R. D. Weld Research Council (New York), Bulletin Series, No. 62, 1960, 28.

Correlation of notch-toughness test results; 37 refs.

Kazinczy, F. de. Jernkont. Ann. (Stockholm), 1959, 143, 617-645.

Dislocation concepts.

Kennedy, R. British Welding J., 1957, 4, 529.

Butt-welded specimens, 3 ft. sq.  $\times$  1 in. thick with central saw-cut notch, suffered brittle fracture at  $+50^{\circ}$  F. to  $-13^{\circ}$  F.; after stress relief, fracture occurred only below  $-58^{\circ}$  F.

Kihara, H., and Masubuchi, K. Welding J. (New York), 1959, 38, 159-168.

Residual welding stresses are influential.

Kller, E. P., and Jensamer, M. Ship Structure Committee (Washington), 1953, Report No. SSC—30.

Fractures in Liberty and Victory ships were due as much to sharp notches as to material. A low transition temperature is all important.

Kochendorfer, A. "Proc. N.P.L. Creep and Fracture Symposium," London, 1956, H.M.S.O. No. 263, 284.

Explanation of brittle and ductile fracture in terms of static and dynamic features of dislocations.

Kochendorfer, A., and Scholl, H. Stahl u. Eisen (Düsseldorf), 1957, 77, 1,006.

Bending test at 190 tons/sq. in./sec. Rapid loading raises transition temperature and increases the multi-axiality of stress.

Kraichik, M. M., and Pashkov, N. V. Svarochnoe Proizvodstvo (Moscow), 1960, 8, 6.

Sensitivity of various steels to welding-burn notches.

Lagasse, P. E., and Marquet, F. Revue Mét. (Paris), Mem. Sci., 1959, 56, 629-639.

Notch effect is different in mild and high strength steels; in routine testing a sharp notch is best.

Leslie, W. G., et al. Trans. Met. Soc. Amer. Inst. Mech. Engineers (New York), 1960, 218, 699.

The notch toughness of  $0\cdot 10\%$  carbon steels, rimmed or killed, is improved by soaking just above the Ae<sub>1</sub>, and cooling in air.

Leeser, D. O., et al. Nuclear Science Abstracts (Oak Ridge, Teanessee), 1960, 14, 1,910.

Irradiation effects on pressure-vessel steels.

Liede, N. G. West of Scotland Iron and Steel Inst., Conference on Brittle Fracture of Steel, May, 1953.

The shipbuilder's viewpoint.

Lihl, F. Verkstoffe u. Korrosion (Mannheim), 1954, 5, 92.

Limitation of tensile tests. A connection exists between weld-contraction and fracture. Grain-refinement improves weldability in thicker sections.

Low, J. R., Jnr. Welding J. (New York), 1952, 31, 253.

Quench-ageing raised the transition temperature rapidly during the first 10 days and then steadily over 3 years.

Lubahn, J. D. Welding J. (New York), 1955, 34, 5188.

In impact tests, the initiating fibrous tearing passes into abrupt cleavage fracture. The decisive factor is the energy absorbed. Initiation energy is independent of temperature.

Lubahn, J. D. Proc. Amer. Soc. Testing Materials, 1958, 58, 678-684

Where the stress gradient is at the notch root and there is notch-congruency, fracture will occur at the same nominal stress.

Main, S. A. Iron and Steel (London), 1957, 30, 365.
A new test.

Majors, H. U.S. Research Reports, 1960, 34.

Cyclic heating about the critical point lowers the transition temperature.

Mosborg, R. J., et al. Welding J. (New York), 1957, 36, 3938. Strakes of tough material arrest fracture.

Mott, N. F. Engineering (London), 1948, 165, 16, 77.
High-velocity crack propagation.

Mott, N. F. J. Iron Steel Inst. (9th Hatfield Memorial Lecture), 1956, 183, 233.

Dislocation theory.

Mylonas, C., et al. Welding J. (New York), 1957, 36, 98.

A running crack will propagate at 5 tons/sq. in., after exhaustion of ductility at the notch root.

Niederberger, R. B. U.S. Research Reports, 1960, 34, 199.

The notch-sensitivity of low carbon Cr-Mo-V steampiping steel depends on stress as well as on rupture time and temperature.

Noren, T. M. Trans. North East Coast Instn. Engineers and Shipbuilders (Newcastle upon Tyne), 1956, 72.

Tensile test pieces, with welded brittle alloy at edges, for the determination of "nominal cleavage strength."

Osgood, W. R., et al. Ship Structure Committee (Washington), Monograph, 1954, Reinhold, New York.

Residual stresses can initiate brittle fracture at lower load, if the steel is either notch-sensitive or below the transition temperature. Most ships' failures have occurred in the deck and bottom shell. Tensile and residual stresses in welds. Butt-fractures have been numerous.

Owen, W. S., et al. Ship Structure Committee (Washington), 1957, Report No. SSC—109.

Fracture at -320° F.; Lüder line microcracks; dislocation pile-up was not confirmed.

Owen, W. S., et al. Welding J. (New York), 1957, 36, 5038—5118.

Charpy values and microstructure of three ship steels.

Owens, D., and Duss, L. U.S. Research Reports, 1960, 34, 324.

Bibliography, prompted by missile and space programmes.

Orawan, E. Reports on Progress in Physics (London), 1948-49, 12, 185.

For the sharpest notch, the stress-concentration factor is 3·5. Brittle behaviour where the yield point exceeds the fracture stress.

Pearson, T. F. Trans. North East Coast Instn. Engineers and Shipbuilders (Newcastle upon Tyne), 1955/6, 72, 157-182, D.53-66.

An account of brittle fracture; production of ship plate; notch-tough and ordinary mild steel compared.

Pellini, W. S. Welding J. (New York), 1956, 35, 2178.

Danger of low notch-ductility welding. E 6010 electrodes are non-ductile at  $-10^{\circ}$  F. to  $+20^{\circ}$  F.; E.12015 and E.10015 at  $-70^{\circ}$  F. to  $+50^{\circ}$  F.

Pigott, E. C. Shipbuilding and Shipping Record (London) International Design and Equipment, 1960, 24–30.

Comprehensive survey of catastrophic brittle fracture: characteristics, mechanism, material, special tests, safeworking temperatures, design, welding, radiography, inspection standards.

Plummer, F. L. Welding J. (New York), 1946, 25, 1,081.
Failure of two natural gas containers in 3½% Ni steel.

Puzak, P. P., et al. Welding J. (New York), 1952, 31, 5618, also 1953, 32, 3018.

Ship-plate fractures are reproducible.

Puzak, et al. Ship Structure Committee (Washington), 1954, Reports No. SSC—77 and SSC—78.

Crack-starter tests; rimmed and semi-killed steels are brittle at low temperatures. Initiation occurs readily where the Charpy value is below 10 ft. lb.; propagation is restricted where the Charpy value is above 15–25 ft. lb. Explosion tests at 20°-60° F. show that only sharp defects (arc-strikes) eliminate deformation.

Puzak, P. P., and Pellini, W. S. Welding J. (New York), 1955, 34, 5778.

The effectiveness of an overlay of notch-tough weld metal at breaks in large welded structures.

Roberts, D. K., and Wells, A. A. Engineering (London), 1954, 178, 820.

Confirmation of Mott's view that the velocity is governed by the supply of kinetic energy to the crack field, and also of Griffith's view that stored elastic energy is supplied.

Robertson, T. S., and Hunt, D. J. W. Scot. Iron Steel Inst., 1952-53, 60, 259.

At a given temperature and for a given material the force for propagation is constant.

Robertson, T. S. J. Iron Steel Inst., 1953, 175, 361.

Since notched test bars are not subjected to previous yield, a more realistic (crack-arresting) test was devised; 1 in. diameter hole;  $\frac{1}{10}$  in. axial saw-cut; catastrophic temperature and stress determined.

Robertson, T. S. Ship Structure Committee (Washington), 1955, Report No. SSC-97.

Critical stress varies inversely with grain size. There is an elastic condition ahead of the crack.

Rolfe, S. T., et al. Welding J. (New York), 1959, 38, 1,695–1,755.

The residual compressive strain yield is influential.

Rudkin, R. J. W. British Welding J., 1956, 3, 22.

In shipbuilding, residual stress may provide energy for propagation.

Sarrak, V. I. Uspeckhi Fiz. Nauk (Moscow), 1959, 57, 339-361 Review

Schaefer, A. O. American Society Mechnical Engineers, Dec. 1957, Paper No. 57—A—262.

Rotor investigation programme.

Shank, M. E. Ship Structure Committee (Washington), 1953, No. SSC-65.

Land-structure failures occurred under static loading and thermal stresses, residual stress being merely contributive. Cold-forming is conducive to brittle fracture, which has complex causes.

Shank, M. E. (Editor). "Control of Steel Construction to Avoid Brittle Fracture," 1957, New York: Welding Research Council.

Text book on materials, fabrication and design for the practical engineer.

Slaughter, E. R., et al. U.S. Research. Report, 1956, 58.
Tidu List 723. (P.B.121357.)

Failure of high-strength steel can occur at 10% of the U.T.S. Composition and structure are not influential.

Soete, W. J. W. Scot. Iron and Steel Inst., 1952-3, 60, 276.
No longitudinal cracks in brittle fracture, as in shear.

Soete, W. "Notch Bar Testing and Its Relation to Welded Construction," 1953, London, Inst. of Welding. Notch stress localisation.

Soete, W. Revue Mét. (Paris), 1954, 51, 813.

Silky fracture occurs at an angle to the principal stress, with marked resistance and distortion. The thickness resistance is 0.3–0.6 of the longitudinal resistance. Necking imposes triaxial stress and splitting.

Srawley, J. E., and Beachem, C. D. Nuclear Science Abstracts (Oak Ridge, Tennessee), 1959, 13, 2,708.

Crack propagation in centre-cracked sheet tensile specimens of low alloy air-hardening steel.

Tipper, C. F. British Welding J. 1956, 3, 435.

Survey 1943–1956. Steels of initial high energyabsorption frequently have a crystalline fracture, while a low-energy absorption may be obtained where there is no trace of cleavage-plane fracture.

Todd, B. Trans. Inst. Marine Engineers (London), 1957, 49, 320.

Brittle fracture of anchor cable and lifting-gear chains.

Tipper, C. F. British Welding J. 1961, 8, 278.

Strain of 4–5% raises the transition temperature by  $20-30^{\circ}$  C.

Turner, C. E. J. Iron Steel Inst., 1961, 197, 131.

Pre-compression at room temperature of notched tensile test pieces eliminates plastic deformation in fractures formed below the transition temperature.

Vanderbeck, R. W. Ship Structure Committee (Washington), 1956, Report No. SSC—101.

Semi-killed 0.15% C, 1.2% Mn open hearth steel as an alternative.

Vanderbeck, R. W. J. Welding (New York), 1951, 30, 598.Significance of impact testing.

Vanderbeck, R. W., et al. American Society Testing Materials (Philadelphia), 1953, Special Tech. Pub., No. 158, 308.
Interpretation of impact test results.

Vasta, J. Soc. Naval Architects and Marine Engineers (New York), 1954.

Claim that aluminium alloys are not notch-sensitive. A monolithic structure is conducive to fracture. Schenectady T-2 tanker cracked in two while being fitted out in 1943. Nearly all subsequent failures occurred in winter months, upon a sudden drop in temperature. Notch-tough materials deform at low temperatures. Nickel and manganese lower the transition temperature. Notch-toughness falls with increasing thickness. Low-hydrogen electrodes promote notchtoughness. Typically,  $2\frac{1}{4}\%$  Ni,  $1\frac{1}{4}\%$  Cr steel had a Charpy value of 40 ft. lb. at  $-40^{\circ}$  F. Sea conditions are influential.

Vedeler, G. Veritas, 1955, 1, 2,

Brittle fracture is liable to occur in supertankers if thick plates are coarse-grained. British prices of finegrained thick plates said to be excessive.

Vedeler, G. Europ. Shipbuild., 1957, 6, 102.

Dimensions and temperature. Fracture can start from sheared edges (but not from machined notches) at a stress much below the yield point; also from hatches and ventilators. Large ships need crack-resistant steel and high-Charpy electrodes.

Veen, J. H. van der. Schip en Werf., 1957, 24, 99,

The most important factor is grain size; the transition temperature rises with increasing thickness of plate.

Warren, W. G., and Vaughan, H. C. Trans. Inst. Welding (London), 1953, 16, 127.

Preheating (100 $^{\circ}$  C.) and Class 6 electrodes recommended for high ductility and crack-resistance.

Weck, R. Trans. Inst. Welding (London), 1959, 13, 41.
Schnadt notch-acuity impact test: ratio of absorbed energy to root diameter.

Wells, A. A. J. W. Scot. Iron Steel Inst. 1952-3, 60, 313. Strain-hardening is an important factor.

Wells, A. A. Welding Research (British Welding Research Association, London), 1953, 7, 34.

Overloading of the notch-root, plastic and elastic residual stress; ships' cracks from butt welds, with transverse spreading.

Wells, A. A. Trans. North East Coast Inst. Engineers and Shipbuilders, 1955, 71, 279.

Effect of size; Docherty effect.

Wells, A. A. Trans. Inst. Naval Architects (London), 1956, 98, 296–326.

Brittle fracture occurs at the critical temperature; complete failure at half the yield-point stress. Weld-contraction stresses are detrimental.

Wells, A. A., et al. British Welding J. 1956, 3, 554.

The larger the specimen, the greater the strain-energy available to propagate a brittle crack. Wells, A. A. British Welding J. 1961, 8, 259.

In small-scale Charpy tests, the fracture appearance is as informative as the impact energy value. Of 60 notched and welded specimens from 1 in. thick low-carbon steel plate, semi-killed or fully killed, 35 fractured either at a low applied stress or without plastic extension. Low hydrogen electrodes are best.

Wessel, E. T. Proc. Amer. Soc. Testing Materials (Philadelphia), 1956, 56, 540-552.

Tests on rimmed steel down to −269° C. Below 100° C., plastic deformation precedes fracture.

Williams, M. L. Ship Structure Committee (Washington), Report No. NBS-6, 1957, (107 pp.).

Increase in carbon or phosphorus content raises the Charpy V-notch transition temperature and makes fracture more probable. Increase in silicon content  $(0-0\cdot12\%)$ , confers finer grain, allows of thinner plate, and lowers the transition temperature.

Williams, M. L. Ship Structure Committee (Washington), Report No. NBS-7, 1957, (43 pp.).

Fracture is initiated where stress-concentration and notch-sensitivity coincide at the operating temperature.

Williams, M. L., and Ellinger, G. A. Welding J. (New York), 1953, 32, 4988.

Fractures in plates from 100 welded ships originated at design notches or welding notches and usually occurred at low temperature. Stress-concentration and notch-sensitivity increased with rising carbon and phosphorus contents, and decreased with rising manganese content.

Winne, H. D., and Wundt, B. M. Amer. Soc. Mechanical Engineers, No. 57—A—249, 1957.

Griffith-Irwin theory; notched rotor discs fractured at 1-3 tons/sq. in, (26-29% of yield strength).

Zeuner, H., and Honer, K. E. Geisserei (Düsseldorf), 1960, 47, 623.

Effect of heat treatment on low-temperature notchtoughness of low alloy steels. High strength at room temperature can be unfavourable. Nickel contents may be adverse. Inclusions are liable to behave as notches.

#### **Availability of Original Papers**

Readers wishing to consult the original papers in technical libraries should experience little difficulty, although for some of the publications a few days' notice may be required. Copies of certain reports and journals are available for purchase. Addresses of the various publishers will be found in a number of directories available in most technical libraries. These publications include the Engineering Index (published annually by Engineering Index Inc. 29th West 39th Street, New York 18, N.Y.), the Applied Science and Technology Index, and the Encyclopædia of American Associations. A list sometimes available in libraries, but now out of print, is the Royal Society's A List of Periodicals and Bulletins containing Abstracts in Great Britain (Cambridge University Press). The Ship Structure Committee Reports may be obtained from the United States National Research Council, Washington D.C.

Translations of a number of the foreign papers are available from the Iron and Steel Institute, 4 Grosvenor Gardens, London, S.W.1. Many of the articles were abstracted in the Journal of this Institute (under an index in each volume and a cumulative index for the period 1951–60) and in the Engineering Index, in which the abstracts more often present investigational results.

#### New and Revised British Standards

NOTCHED BAR TESTS: PART I: THE IZOD IMPACT TEST ON METALS (B.S.131: PART I: 1961) PRICE 6s.

THIS British Standard, which deals with the Izod impact test on metals, is published as Part I of a revision of B.S.131. (Part II, published in 1959, deals with the Charpy V-notch test). The scope of the standard has been widened to include details of the method of test with structural and dimensional requirements for the testing machine. Whereas the former edition of the standard gave only nominal dimensions of test pieces, the new edition gives dimensions with appropriate machining tolerances. The tolerances for ferrous test pieces are in accordance with a recommendation of the International Organisation for Standardisation, but for non-ferrous metals a more restrictive tolerance has been applied to the dimensions of the test piece. The structural and dimensional aspects of the testing machine are fully dealt with and an appendix gives recommendations for its installation.

ZINC COATINGS ON IRON AND STEEL ARTICLES (B.S.729: 1961)

PART I: HOT DIP GALVANISED COATINGS. PRICE 4s.
PART II: SHERARDISED COATINGS. PRICE 3s.

The revised edition of B.S.729 has been divided into two parts to cover hot dip galvanised coatings and sherardised coatings, respectively. The earlier standard specified visual examination and Preece copper sulphate dip continuity test for coatings. The revised standard introduces requirements, in Part I, for weight of coating as well, giving three alternative methods of determination. Guidance is given on threaded work and an accepted procedure indicated. In Part II, visual examination and the copper sulphate dip test are alone specified. Again, recommendations for threaded work are included.

CAST IRON BATHS (B.S.1189: 1961). PRICE 6s.

ONE of the hazards of old age or sickness for many people is that, having once got into their bath, they have difficulty in getting out again. From time to time, old folk, trying to put one leg over the rim of the bath, have slipped and injured themselves. In response to a request from those responsible for the care of elderly people and invalids, the B.S.I. technical committee which recently revised the British Standard for cast iron baths (B.S.1189) therefore added a rectangular, shallow bath to the existing range of patterns. It is hoped that in future, this type of bath will become a standard fixture in old people's residential and nursing homes, and in dwellings built specially for the elderly.

The new pattern is as shallow as possible, while allowing for the normal height for fixing a trap under the bath above floor level. Where it is possible to sink the trap below floor level, the rim of the bath can then be brought to only 16 in, above floor level. In this type of bath, it is also possible to have the tapholes in a position allowing cornerwise fixing near the front of the bath—an added convenience for the elderly. When ordered, raised handgrips may also be fitted. As there is now a growing tendency not to fit overflows to baths, except where by-laws insist on this, the provision of

holes for overflow fittings has been made optional. Among other changes in the standard is the inclusion of advice on filling and cleaning baths.

DETERMINATION OF AIR POLLUTION (B.S.1747: 1961)

PART I: SPECIFICATIONS FOR DEPOSIT GAUGES.
PRICE 58.

When B.S.1747 was first published in 1951 it was concerned solely with deposit gauges. Now it is being reissued in several parts to cover, in addition, apparatus and procedure for the measurement of (1) fine particles (smoke), and (2) gaseous impurities, as well as the solid material (such as grit) which is measured by the deposit gauges specified in Part I of the standard. Experience in the use of deposit gauges during the past ten years has enabled the British Standards Institution committee concerned to introduce a number of refinements into the standard, but the procedure outlined is substantially unchanged. The standard deals with the construction, installation and use of deposit gauges. A standard report form and a statement of the analytical determinations to be made are included.

Reference Tables for Thermocouples (Copper v Constantan) (B.S.1828: 1961). Price 68.

THE British Standards Institution has published another set of reference tables for the purpose of converting thermocouple voltages into equivalent temperatures. The new tables are published as B.S.1828: Reference Tables for Thermocouples (Copper v Constantan). Like the other reference tables in this series (B.S.1826 and 1827), they are based on the International Temperature Scale 1948 and on the Absolute Volt which, in 1948. was adopted as the standard unit in place of the International Volt. The electromotive force/temperature curve adopted as a basis for the tables is one typical of the wire supplied by British manufacturers, since, after extensive research, it has been found impracticable for British wire manufacturers to produce copper-nickel alloy in bulk which will give values in accord with the tables of the United States National Bureau of Standards.

ELECTROPLATED COATINGS ON THREADED COMPONENTS. (B.S.3382: PARTS I AND II: 1961) PRICE 7s. 6d.

PART I: CADMIUM ON STEEL COMPONENTS.

PART II: ZINC ON STEEL COMPONENTS.

Parts I and II of a new British Standard dealing with the plating of small threaded components such as bolts, screws and nuts, have been published as one booklet (B.S.3382: Parts I and II). They deal with thicknesses of plating which, without preventing the assembly of threaded components, will ensure satisfactory protection against corrosion. Introductory notes explain the problems associated with the electroplating of threaded components and the need for special procedures for thickness specification, inspection sampling and thickness determination.

As well as specifying plating thicknesses, sampling and other inspection procedures, and methods of thickness determination, this publication lays down requirements for the purity of the plating, for reduction of hydrogen embrittlement, for finish and appearance of plated surfaces, and for passivation. In the case of cadmium plating, requirements are also laid down for adhesion and for freedom from gross porosity. Tables of surface areas of standard screws, bolts and nuts are given as a guide to electroplaters, so that they may estimate the conditions of current and plating time needed to obtain the required deposit thickness, and also to assist in the estimation of average thickness on

plated components.

The plating thicknesses laid down are intended for components having threads of basic major diameters from 0.060 in. to \$\frac{1}{2}\$ in., inclusive, specified in B.S.84, 93, 811, 1580, 1095 and 2779. The publication refers in particular to the general purpose bolts, screws and nuts specified in B.S.57, 450, 1083, 1768, 1981 and 3155. The plating thicknesses specified will be adequate for most purposes but under severe conditions, greater protection may be needed. In such cases, reference should be made to Part VII, B.S.3382, to be published in due course under the title, "Thicker Plating for Steel Components." Other parts will deal with nickel and nickel/chromium on steel components, nickel and nickel/chromium on brass components, tin on brass components and silver on brass components.

Dosemeters for Protecting Workers from Radiation (B.S.3385). Price 3s.

At a time when X- and gamma rays are being increasingly used as aids to medicine, industry and research of all kinds, B.S.3385 will be welcomed by all those responsible for the health and safety of workers exposed to radiation hazards. This new British Standard lays down requirements for direct reading personal dosemeters for X- and gamma radiation. These instruments—a common type of which looks like a fountain pen—fit into the pocket. They tell the user the amount of radiation dose he has received—if any. Obviously, it is essential that these instruments should be robust and accurate and the requirements of B.S.3385 are intended to ensure that they are.

A note in the foreword draws attention to the effect of beta radiation on measurements of gamma radiation dose and recommends that when the dosemeter is used in the presence of beta radiation, an appropriate sleeve should be fitted to the instrument. It also points out that unsealed instruments may be seriously affected by large changes in humidity, changes in atmospheric pressure, and by the presence of significant concentrations of radioactive or ionised gases. In such circumstances, it is recommended that a sealed instrument should be used. A companion standard, for film badges,

is in course of preparation.

B.S.3385 is the first British Standard to be prepared under the authority of the newly-formed Nuclear Energy Industry Standards Committee of the British Standards Institution. Under the direction of this committee, several technical committees are at work, dealing with nuclear terminology, units and symbols, protection against ionising radiation, nuclear reactor safety and radioisotopes. Almost all their work is being done in close consultation with the International Organisation for Standardisation (ISO) since it is regarded as of paramount importance that nations should be uniform in their standards in the field of nuclear energy. It is, for instance, obviously vital that a symbol on a package, denoting that the contents are radioactive, should be understood in all the countries through

which that package may travel. Recommendations for a warning symbol, based on draft ISO proposals, are being prepared by a B.S.I. technical committee. This symbol would appear wherever there was a radiation danger: on storage cabinets containing radioactive materials, on doors of X-ray rooms, on packages of radioactive materials, and so on.

Aluminium Food Storage Canisters (B.S.3394).
Price 3s.

Culinary Bailing Bowls (B.S.2974). Price 3s. Aluminium Pudding Basins (B.S.3390). Price 3s. Tinned Steel Baking Dishes (B.S.3393). Price 5s.

Bun Tins (B.S.3392). Price 3s.
Aluminium Pir Dishes (B.S.3391). Price 3s.

THESE British Standards are six in a series for culinary equipment which is being prepared to meet the needs of Her Majesty's Forces, Government purchasing departments, local authorities and other large organisations which purchase in bulk. Dimensions as well as manufacturing requirements are laid down. Equipment conforming to these standards will be of sound quality and should wear well. Manufacturers may therefore think it worthwhile to work to these standards when

Copies of these Standards may be obtained from the British Standards Institution, Sales Branch, 2 Park Street, London, W.1. (Postage will be charged extra to non-subscribers.)

making equipment for the general public.

#### **Nuclear Power Station Heat Exchanger**

The first of the two heat exchangers to be manufactured by Jönköpings Mekaniska Verkstad for the Ågesta nuclear power and heating plant, outside Stockholm, is now being installed. Designed in close collaboration with the customer, the Swedish Atomic Energy Company, the units are to operate between the reactor's primary cooling system, containing heavy water, and the secondary light-water system. The temperature on the heavy-water side is 220° C. and the operational pressure 33 atmospheres, which is transformed into saturated steam of 14 atmospheres on the light-water side. Each exchanger contains 41,000 ft. of Sefab (Sandvik) stainless-steel tubes. X-ray, ultrasonics and microscopy have been extensively used for inspection throughout the manufacturing process, and before delivery the completed unit was submitted to a helium test to ensure that it was absolutely leak-proof.

#### **New Staveley House Journal**

A NEW house paper, the Staveley Express, recently went out to the 8,000 employees of Staveley Industries, Ltd., whose twenty-five companies are spread throughout Britain. Copies were also despatched to Staveley staff in Vancouver and Toronto. The brightly illustrated four-page paper is printed in two colours and contains features on the group's important role in Britain's fight for exports, the £500,000 order for dust-collecting plant recently signed in Moscow by one of the member companies, and a picture competition for employees with £90 in prizes. It also includes articles on industrial safety, home decorating with the group's own products, and several other items of general interest to Staveley staff.





# NEWS AND ANNOUNCEMENTS

#### International Beryllium Metallurgy Conference

An international conference on "The Metallurgy of Beryllium," arranged by the Nuclear Energy Committee of the Institute of Metals, will be held at The Royal Commonwealth Society, Craven Street, London, W.C.2, between 16th and 18th October, 1961. There is a large mass of hitherto unpublished work on beryllium, and the purpose of the conference is to publish and discuss this work, which covers the whole field of the metallurgy of beryllium, with the exception of the extraction processes. Seventy papers have been submitted for discussion and these have been grouped under four main headings, namely: mechanical and physical properties: beryllium in nuclear reactors: beryllium in aircraft. and metal preparation and fabrication. Within these headings the papers have been further subdivided for discussion purposes: the papers comprising each subgroup will be presented by a rapporteur.

All persons registering for the conference, for which a registration fee of £12 will be charged, will receive a full set of preprints of all the papers submitted. A bound volume of the proceedings (refereed and edited papers and discussion) will be published as soon as possible after the conference. Further particulars may be obtained from the Secretary, The Institute of Metals, 17 Belgrave Square, London, S.W.1.

#### Greek Blast Furnace Lining Order

In the face of strong competition from German and Japanese companies, Carblox, Ltd., the carbon brick manufacturing company of the Marshall Refractories group of Sheffield, have secured an order for a lining for a new blast furnace to be built near Athens. Carblox will also supply the firebrick linings and checker fillings for the two hot blast stoves which will be built for operation with the furnace. This new plant-the first blast furnace in Greece-will be built by Acieries Halivourgiki S.A., of Athens, who at the moment have only a steel works. It will use iron ore from sources in Greece and thus save the importation of pig iron. This £100,000 order represents a further substantial addition to the export business of the Marshall Group, which now sells refractories to thirty-six different countries.

#### Summer School at Cambridge

Scientists from many parts of the world who visited the laboratories and works of the Cambridge Instrument Company on Tuesday 25th July saw several Microscan X-ray analysers in various stages of construction and examined the greatly expanded production facilities for dealing with current Microscan orders. The Microscan is perhaps the most successful commercial X-ray microanalyser in full production-sixteen having been installed within the past eighteen months in British. American. Dutch and German research and development organisations.

The visitors were delegates from a summer school on X-ray microanalysis which was held in Cambridge, from 24th July to 4th August, under the auspices of the University Board of Extra-Mural Studies. The School was convened by Dr. V. E. Cosslett of the Cavendish Laboratory, who has directed important fundamental work in X-ray microanalysis and is well known for his research into the principles and applications of both

X-ray analysis and electron-microscopy

In addition to the visit to the Cambridge Instrument Company the agenda included a visit to Tube Investment Research Laboratories, opportunities for practical work. lectures describing the development, applications and general principles of the technique, and surveys of commercially available instruments. Lecturers included, Dr. Cosslett, Professor R. Castaing of France, the originator of the technique, Mr. T. Mulvey of AEI Research Laboratories, Professor R. E. Olgivie of Msasachusetts Institute of Technology, Drs. D. A. Melford and P. Duncomb of Tube Investments Research Laboratories, and Dr. Nixon of the University Engineering Laboratory.

#### Physical Metallurgy Refresher Course

A REFRESHER course on "Modern Concepts in Physical Metallurgy" is to be held in the Department of Metallurgy at the University of Manchester. It is intended for metallurgists and other scientists working in the field who are graduates or Associates of the Institution of Metallurgists. The ten lectures will be given by members of the staff of the department on successive Monday evenings, commencing on 2nd October, 1961. and will deal with modern concepts in the field of electron theory, dislocations and their applications in physical metallurgy. Each lecture will be of roughly one hour duration, followed by half an hour tutorial. Fee for the course is five guineas: further particulars can be obtained from the Director of Extra-Mural Studies, The University, Manchester, 13.

#### Polarographic Society Meeting

A MEETING of the Polarographic Society will be held on Wednesday, 25th October, 1961, at 2.45 p.m. at the School of Pharmacy, 29-39 Brunswick Square, London, W.C.1. Prof. W. H. Linnell has agreed to take the chair, and Prof. Dr. M. von Stackelberg, of Bonn University. will read a paper (in English) entitled: "Some Special Problems in the Polarography of Indium and Tellurium." At this meeting, Prof. von Stackelberg will be presented with the Society's Medal for the current year.

#### New Wellman Subsidiary Companies

THE WELLMAN SMITH OWEN ENGINEERING CORPORA-TION, LTD., announce that due to the expansion of their activities they have decided to segregate and operate the sales organisations of their divisions dealing respectively with cranes and mechanical handling equipment and rolling mill equipment as separate subsidiary companies under the titles of Wellman Crane and Machine Co., Ltd., and Wellman Rolling Mills, Ltd.-both of Parnell House, Wilton Road, London, S.W.1.

MR. J. N. REES, general sales manager (mechanical division) of the Corporation, has been appointed managing director of Wellman Crane and Machine Co., Ltd., the board of which also includes Mr. V. Bellingham, MR. D. E. POPE, MR. J. H. KNIGHT, MR. W. R. ROBERT- SON, MR. S. W. TAYLOR and MR. E. H. DICKSON. The board of Wellman Rolling Mills, Ltd., will consist of MR. C. BROOKS (managing director), MR. V. BELLINGHAM, MR. G. W. CLAMP, MR. R. CURDIE, MR. D. H. GOODREID, MR. R. C. LEES and MR. A. V. MCCUMISKEY.

The operations of these subsidiary companies will be supported by the full engineering and manufacturing resources of the Wellman Group for the design and construction of cranes and handling machinery for the metallurgical and other industries, and for the design and construction of rolling mill equipment, including continuous pickle lines, strip processing lines, etc., and also seamless tube making plants.

#### I.S.I. Autumn Meeting

"The Future of Ironmaking in the Blast-furnace" is the title of the symposium that forms the major technical subject of the Iron and Steel Institute's autumn meeting. Iron-making experts from Belgium, France, the U.K., and the U.S.A., have contributed papers for the four sessions, which cover injection processes, hot-blast stoves, burden preparation, and furnace engineering, respectively.

In addition, there will be a series of parallel scientific sessions, devoted to the thermodynamics of slags, oxidation and scale, bainite, and the effect of steel-making practice on the mechanical properties of alloy steels.

The Iron and Steel Engineers' Group has been responsible for organising a session on the effect of the various steelmaking processes on the energy balance of integrated iron and steelworks, which will follow the symposium on ironmaking.

The symposium on ironmaking and the session on the energy balance of integrated iron and steelworks will be held at the Federation of British Industries, Tothill Street, London, S.W.1, on 29th to 30th November and on 1st December, respectively. The scientific sessions will take place at the offices of the Institute, 4 Grosvenor Gardens, London, S.W.1, on 29th to 30th November.

#### **Nuclear Developments Board**

NUCLEAR DEVELOPMENTS, LTD., a private company recently formed by Imperial Chemical Industries, Ltd. (Metals Division), Rolls-Royce, Ltd., and The Rio Tinto Co., Ltd., has appointed the following board of directors: Mr. J. N. V. Duncan, O.B.E. (managing director, Rio Tinto) who will be chairman, MR. R. W. WRIGHT (deputy managing director, Rio Tinto), SIR MARK TURNER (director, Rio Tinto), Mr. J. D. PEARSON (chief executive and deputy chairman, Rolls-Royce), Mr. A. A. RUBBRA, C.B.E. (director, Rolls-Royce), Mr. L. Barman (special executive, Rolls-Royce), Dr. J. TAYLOR, M.B.E. (director, I.C.I.), Mr. St. J. DE H. ELSTUB, C.B.E. (chairman, I.C.I. Metals Division), and Dr. R. L. P. Berry (director, I.C.I. Metals Division). The executive director and general manager will be Mr. S. S. SMITH (research manager, I.C.I. Metals Division), who has been closely associated with nuclear engineering since 1941, when he made important contributions to the development of diffusion membranes for isotope separation. He has since been continuously concerned with research and development work on specialised nuclear engineering materials. Mr. P. J. G. ELWES (Rio Tinto)

will be secretary. Nuclear Developments, Ltd., has been formed to process and manufacture nuclear fuel materials and to fabricate reactor fuel elements.

#### Lecture Course

A COURSE of ten lectures on "Applied Physical Chemistry for Metallurgists," organised by the Metallurgy Department of the Battersea College of Technology, will be given by Dr. J. Mackowiak at 6.30 p.m. on successive Wednesdays, commencing 11th October, 1961. The course has been designed to meet the needs of those employed on research and production who either completed formal studies some years ago, or whose professional training did not include any previous study of the subject. It is intended that the course, whilst complete in itself, will also serve as a preliminary for further courses on specific topics. The syllabus will be divided into four main sections, namely electron theory of elements and molecules; thermodynamics; kinetics; and application to practical problems. The fee for the course is £1 and enrolment forms may be obtained from the Secretary (Metallurgy Courses), Battersea College of Technology, London, S.W.11.

#### Powder Metallurgy Joint Group Meeting

The winter meeting of the Powder Metallurgy Joint Group of The Iron and Steel Institute and The Institute of Metals will be held in the Hoare Memorial Hall, Church House, London, S.W.1, on 7th and 8th, December 1961. The main discussion will be the symposium on "Sintered High Temperature Compounds" on 8th December, between 10 a.m. and 5 p.m. The papers comprising the symposium will all be published in Powder Metallurgy No. 8. Copies of this and further details of the Joint Group and its meetings are obtainable from the Secretary of the Powder Metallurgy Joint Group, 17 Belgrave Square, London, S.W.1.

#### Institution of Metallurgists' Examinations

The Institution of Metallurgists has announced the results of the examination held in July last at fourteen examination centres in the U.K. and twelve centres abroad. Of the 341 candidates for the Associateship, 142 passed, 119 were referred in one subject, and the remaining 80 failed. In the examination for the Licentiateship 196 candidates passed, 132 were referred and 403 failed. The pass lists will be published in full in the Institution's journal, The Metallurgist.

#### B.S.A. Tools - Metachemical Link-Up

B.S.A. Tools, Ltd., of Birmingham and Metachemical Processes, Ltd., of Crawley, have formed—on an equal partnership basis—a new company, Metachemical Machines, Ltd., which will utilise all available technical knowledge through a worldwide series of agreements in the production and development of equipment for electrochemical machining which will be sold both in Britain and overseas. The technique of electrochemical machining originated in research sponsored by the Steel Improvement & Forge Co. (SIFCO), of Cleveland, Ohio,

and technical information from SIFCO will be available to Metachemical Machines, Ltd.

Scientific work for the new company will be carried out at its Crawley headquarters and production will be undertaken initially by the Kemworthy Jig & Press Tool Co., Ltd., of Morden, which has been acquired by Metachemical Machines as a wholly owned subsidiary. On the home market the new company's products will be sold by Burton, Griffiths & Co., Ltd., of Kitts Green, Birmingham, the U.K. selling organisation of the B.S.A. Tools Group. Sales in overseas markets will be organised through the existing export network of B.S.A. Tools, Ltd.

#### Conference on Hydrogen in Steel

At the conference on "Hydrogen in Steel," which is to be held at Harrogate from 11th to 13th October, under the auspices of the British Iron and Steel Research Association, the opening lecture will be given by Professor A. R. Troiano, of the Case Institute of Technology, Cleveland, U.S.A., who will deal with the role of hydrogen in the mechanical behaviour of metals.

The programme includes five other subjects for papers and discussions, namely: the removal of hydrogen from liquid and solid steel; the effect of hydrogen on the properties of ultra high tensile steels; the diffusion and solubility of hydrogen in steel; hydrogen in weld metal; and the unknown—unresolved factors about hydrogen in steel. Full details of the arrangements, together with application forms, can be obtained from the Technical Secretary, Metallurgy Division, The British Iron and Steel Research Association, 11 Park Lane, London, W.1.

#### Iron and Steel Institute Officers

The council of the Iron and Steel Institute have decided to nominate Mr. M. A. Fiennes, group managing director of Davy-Ashmore, Ltd., at the Institute's autumn general meeting on 29th November, 1961, for election at the annual general meeting on 2nd May, 1962, as president for 1962-63.

At the council meeting on 26th July, 1961, Mr. H. W. A. Waring, C.M.G., general managing director and deputy chairman of GKN Steel Co., Ltd., was elected honorary treasurer of the Institute, in succession to Sir Julian Pode, deputy chairman and managing director of the Steel Company of Wales, Ltd. Sir Julian, who had served as honorary treasurer since 1959, was elected a vice-president of the Institute.

#### Hedin Reorganisation

Hedin, Ltd., of South Woodford, manufacturers of industrial electric heating equipment, have announced that as a result of continued expansion of their business they have taken over additional office and works accommodation at Fowler Road, Hainault, Essex. The manufacture of their industrial heating elements and resistances will continue to be carried out at South Woodford, whilst furnaces and ovens will be made at Hainault.

Although the company was formed in 1930, it is only in the last six or seven years, under the direction of Mr. J. ROYCE, that considerable progress has been made in the development and manufacture of electric furnaces. To enable Mr. Royce to devote even more time to re-

search and development work, Mr. D. Hobson has been appointed sales manager of the furnace division. Mr. Hobson, before joining Hedin, was assistant sales manager with Salem Brosius (England), Ltd. The sales of industrial ovens will continue to be handled under the managership of Mr. P. Keen.

#### Personal News

Mr. C. F. Hurst has been appointed assistant managing director of Samuel Osborn & Co., Ltd. Mr. Hurst is now deputy chairman and assistant managing director of the company.

ASSOCIATED ELECTRICAL INDUSTRIES, LTD., has appointed a director of research. He is Mr. L. J. Davies, who was director of research of A.E.I. (Rugby), Ltd., in which appointment he is succeeded by Dr. J. E. Stanworth. Mr. Davies becomes responsible for the direct supervision of the four A.E.I. Research establishments at Aldermaston, Harlow, Manchester and Rugby. He reports to the A.E.I. board through Sir Cecil Dannatt, the vice-chairman, and now becomes a non-executive director of A.E.I. (Rugby), Ltd.

The following new appointments are announced by the power plant division of The General Electric Co., Ltd., Erith, Kent, with responsibility to the divisional manager, Mr. D. M. Smith. The former sales manager, Mr. F. L. Tombs, has been appointed manager, engineering services, with executive responsibilities for sales, tendering, contracts and operational services departments and for site resident engineers, and Dr. D. Kalderon, formerly chief development engineer, has been appointed deputy chief engineer. Mr. W. G. Rhodes is now manager, home sales, and Mr. D. C. Thorpe, manager, overseas sales.

With a view to ensuring even more effective representation in England and Wales, some of the area representation of Wild-Barfield Electric Furnaces, Ltd., has been re-arranged. The areas affected, in which the personnel will also represent the furnace division of G.W.B. Furnaces, Ltd. are: Birmingham and East Midlands, where the area manager, Mr. G. W. Haines, will have as sales engineers Mr. R. E. Butchers and Mr. C. A. McNeill; West Midlands, with Mr. A. V. Skelsey as sales engineer; S. Wales, with Mr. T. M. Morgan as sales engineer; Sheffield and North Midlands, where the area manager, Mr. E. J. Heiser will have as sales engineers Mr. D. N. Greensmith and Mr. D. J. Sutherland; Northern England, with Mr. R. Flanagan as sales engineer.

The following appointments are announced by Vickers-Armstrongs (Engineers), Ltd., with effect from October 1st, 1961: Mr. R. F. W. Keay to be an additional member of the board and director of production engineering; Mr. P. D. Scott Maxwell, D.S.C., to be a special director and deputy general manager, Barrow works; and Mr. J. Hay to be a special director and works manager, Barrow works.

A NUMBER of changes in the organisation of Cooke, Troughton & Simms, Ltd., will become effective at October 1st, 1961: Mr. P. D. Scott Maxwell, D.S.C., relinquishes his appointment as managing director on taking up an appointment with Vickers-Armstrongs (Engineers), Ltd., but retains his seat on the board. He

also relinquishes his appointments as chairman and director of C. Baker Instruments, Ltd.; director of Cooke, Troughton & Simms South Africa (Proprietary), Ltd.; president and director of Cooke, Troughton & Simms Incorporated; and director of Casella (Electronics), Ltd. Mr. H. Wright is appointed managing director and takes over the other offices relinquished by Mr. Scott Maxwell with the exception of the last named. Mr. A. J. Munro is appointed engineering director and will be responsible for the technical departments in addition to production: he is also appointed a director of Cooke, Troughton & Simms Incorporated. MR. E. E. KENNAIRD is appointed a director of Casella (Electronics), Ltd., and MR. E. Cussans, works manager of Cooke, Troughton & Simms, Ltd., whilst Mr. S. S. L. MARSHALL becomes commercial manager of Cooke, Troughton & Simms, Ltd., in addition to his existing appointment as secretary.

MR. F. D. STICKLAND, who is at present commercial director, will retire from service with Vickers-Armstrongs (Shipbuilders), Ltd., on March 31st, 1962, having then attained normal retirement age. He will be succeeded by MR. W. A. Till, at present commercial manager, Vickers-Armstrongs (Engineers), Ltd., Barrow works, who will relinquish his present office on December 31st, 1961. Mr. W. J. Scott will relinquish the office of commercial director, George Mann & Co., Ltd., and his seat on the board of that company, on September 30th, 1961, and will become deputy commercial manager, Vickers-Armstrongs (Engineers), Ltd., Barrow works, until he succeeds Mr. Till as special director and commercial manager on January 1st, 1962. Mr. W. J. R. CUPPLES will succeed Mr. Scott as commercial director, George Mann & Co., Ltd., with effect from October 1st, 1961, retaining his present appointments with Vickers-Armstrongs (Tractors), Ltd., Vickers-Armstrongs (Tractor Sales), Ltd., and Vickers-Armstrongs (Tractors) Canada, Ltd.

The George Cohen 600 Group, Ltd., announce the retirement of Mr. C. E. G. Nye, M.C., from the board of George Cohen Sons and Co., Ltd. Mr. Nye, who has been with the company for forty years became manager of the group publicity department in 1931, and in 1946 he was appointed a special director of George Cohen's and ten years later, when the holding company was formed, was elected a full director of George Cohen Sons & Co., Ltd., its largest subsidiary.

THE UNITED STEEL COS., LTD. announce that Mr. N. D. MACDONALD, general works manager of their Workington Iron and Steel Co. branch, has been appointed a director of that branch.

Mr. E. A. Shipley, who joined British Ropes, Ltd. two years ago as head of research of the wire division, has recently been elected a Fellow of the Institution of Metallurgists.

BRIGADIER A. LEVESLEY, O.B.E., M.C., T.D., a director of Edgar Allen & Co., Ltd., has accepted the chairmanship of the Sheffield Standing Conference of Voluntary Youth Organisations in succession to the former Chief Constable of Sheffield, Mr. G. E. SCOTT, O.B.E.

THE U.K. Atomic Energy Authority has agreed to release Mr. P. T. FLETCHER (previously deputy managing director, development and engineering group at Risley and, since April, special adviser to the member

for reactors) to take up the appointment, as from October 1st, 1961, of director, responsible to the board of the United Power Co., Ltd., for construction and supply. United Power Co., Ltd. is a company recently formed to unite the nuclear engineering activities of Atomic Power Constructions, Ltd., and the G.E.C. and Simon-Carves Atomic Energy Group.

DR. E. R. HENSEL, internationally known expert in metallurgy and research administration, and until recently vice-president of engineering of P. R. Mallory & Co., Inc., Indianapolis, Indiana, has joined Clyde Williams & Co., Columbus, Ohio, and its associated technical. management service, and financial enterprises. In the new position, Dr. Hensel will be: president of the Clyde Williams Corporation, an international company providing research, technical information, investment, advisory, and management services in the United States and Europe; and executive vice-president of Clyde Williams & Co., the parent organisation to the various subsidiary operations of the firm. In his role as president of the Clyde Williams Corporation. Dr. Hensel will direct and co-ordinate the activities of the Corporation's international staff and will head the American office.

Mr. P. L. McIlmoyde has been appointed assistant manager, purchasing department of Castrol, Ltd.

AFTER twenty-six years' service with The General Electric Co., Ltd., Mr. J. P. CLIFTON, manager, technical manuals department at the Witton works of the company, retired at the end of July, but will continue to serve in a consultative capacity until the end of September.

The Incandescent group of companies has appointed Mr. W. S. Sinclair to be manager of its Cardiff office, as of the beginning of August. Mr. Sinclair, who has twelve years' service with the company, is already well known in the South Wales area.

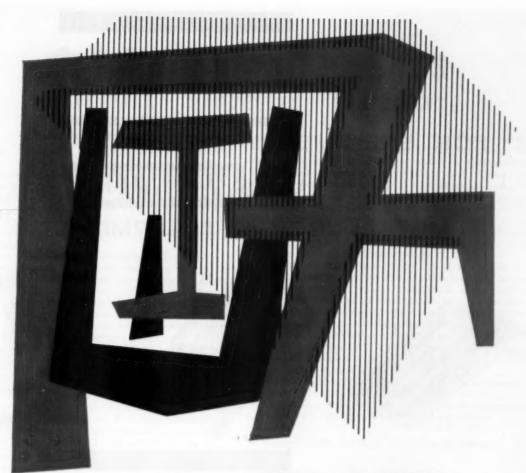
The Rt. Hon. The Earl of Halsbury has been elected president of the Machine Tool Industry Research Association. Lord Halsbury is well known for his work on the application of scientific research, and has many connections with the machine tool industry.

Dr. I. Jenkins has accepted an appointment as director of metallurgical research with The Manganese Bronze & Brass Co., Ltd. and their subsidiaries. He will take up this position from November 1st, 1961.

Mr. L. Holdbrook has joined West Instrument, Ltd. and will represent the company in N.E. London, Essex, Suffolk, Cambs., and Norfolk, working from the company's London office at Granville House, 132/135 Sloane Street, S.W.1. (Tel: SLOane 2191).

MR. L. A. FLETCHER has been elected a Fellow of the Institute of Linguists. Mr. Fletcher is editor of *Metal Powder Report*, a monthly journal of abstracts published by Powder Metallurgy, Ltd., a subsidiary of F. W. Berk & Co., Ltd., Berk House, Baker Street, London, W.1.

MR. H. DIXON, chief inspector at Hoover (Washing Machines), Ltd., Pentrebach, and MR. J. Powell, an overseas approval engineer at Hoover, Ltd., Perivale, have recently completed twenty-five years' of service with the company. In token of this long service they were recently presented with gold watches by Mr. H. G. Meads, comptroller of Hoover, Ltd.



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# RECENT DEVELOPMENTS

MATERIALS : PROCESSES : EQUIPMENT

#### Unitrace and Duotrace

IMPERIAL ALUMINIUM Co., LTD., are now marketing Unitrace tube in the United Kingdom. Unitrace, manufactured by Aluminum Co. of America, is designed for use in the processing of chemical and food products, where liquids—for example, tar, oil, fat, chocolate—must be kept warm to prevent solidifying when being pumped from one location to another. It is equally effective where liquids have to be kept cool at a controlled

temperature.

Unitrace is an extruded aluminium alloy tube combining two integral channels, the larger for the product line, the smaller for the tracer line carrying the heating or cooling medium. Unitrace is light and easily bent but has considerable strength and the integral construction provides excellent thermal characteristics. It is supplied in standard 30 ft. lengths and in seven standard sizes from 1 in. to 8 in. Outside diameter of the tube conforms to standard pipe sizes so that normal insulation can be fitted. Special flange fittings enable Unitrace lengths to be bolted together by standard flanges or to jacketed fittings by means of adaptor flanges. Trace caps enable welded joints to be made on straight runs, tees and cross-connections, and right-angle elbows having trace lines at the side, outside or inside, are available. The range has recently been increased by the addition

Imperial Aluminium Co., Ltd., P.O. Box 216, Witton, Birmingham, 6.

of Duotrace, with two tracer lines instead of one.

#### **Corrosion Rate Measurement**

The Corrosometer, marketed in Great Britain and the Commonwealth by Winston Electronics, Ltd., on behalf of the Crest Investment Co. of Santa Fe Springs, California, United States, is described as "creating a new standard in the measurement of corrosion in the laboratory, process and storage plants in very many industries by giving a swift direct reading of corrosion rate in less than a minute." By the use of such an instrument the efficiency of anti-corrosion methods may be ascertained, and also remedial action may be carried out at the appropriate time, thus eliminating accidents, damage to plant, and hold-ups.

The instrument is based on the decrease of electrical conductivity of a piece of metal which occurs when its cross-sectional area is reduced by corrosion, whether or not the corrosion product is removed from the surface. By means of its systems of probes one instrument, which can be mains- or battery-operated, can measure the corrosion at a number of points. Several hundred feet of extension cable may be used between the probes and the meter in a convenient control room; alternatively the meter can be used as a portable instrument for touring the plant and taking readings regularly at

probe points.

The probes consist of an exposed specimen of the material of which the pipeline, reaction chamber, storage tank, or laboratory sample is made, a reference specimen, and a check specimen. The reference probe is protected from corrosion by a coating which may be epoxy resin for low temperature conditions, fluorocarbon for medium temperature conditions, and ceramic for high temperature conditions. The readings are independent of temperature, since it is the resistance ratio between the exposed specimen and companion coated reference specimen that is measured. The short check specimen, also coated for protection, is compared with the protective coating remains intact.

The meter dial reads directly in micro-inches or micro-centimetres, and no cleaning, weighing or analysis of the specimen is required. Because the instrument is highly sensitive, frequent readings can be made, so that the effect of the process variables or corrosion inhibitors on corrosion rate can be determined in hours and days rather than in weeks and months. The measurement is made while the process is in full operation, because once the probe is installed it need not be removed for months, or even years, if corrosion is carefully inhibited.

The Corrosometer method combines the developments and long-term tests of both the Standard Oil Co., Inc., and Crest Instrument Co., and embodies the principles of Standard's U.S. Patent 2.735.754 under which Cresta

is licensed.

Winston Electronics, Ltd., Shepperton, Middlesex.

#### Continuous-Check Flame Safeguard

A FLAME safeguard unit that checks its own complete circuit once every second is announced by Honeywell Controls, Ltd. It is the first continuous-check flame safeguard unit available in the United Kingdom. A self-checking circuit disconnects the flame rod and checks all components every second. Such checking has hitherto been incorporated in combustion safeguards during start-up or re-cycling only. The new type, known as the R 4075 Protectoglo, will cause immediate shut down and sound an alarm if the sensing signal, amplifier or related circuitry fails at any time while burners are operating. The R 4075A Protectoglo is additional to the W613 type announced earlier this year.

Honeywell Controls, Ltd., Greenford, Middlesex.

#### Preumatic Control System for Batch Processes

A NEW two-term pneumatic process control unit for use on batch processes, is announced by Honeywell Controls, Ltd. Designed to counteract "overshoot" on start-up, it is known as the Batch Air-O-Line and is suitable for use with electronic strip chart recorders and pressure gauges. With existing proportional-plus-reset pneumatic control devices, when the recording pen is outside the proportional band, accumulation of air in the system during the shut-down procedure will cause overshoot on start-up. This gives rise to delays in processing and

endangers quality. The Batch Air-O-Line will reduce or prevent overshoot by automatically discharging accumulated air when the pen moves out of the

proportional band.

When the process is shut down, the controller output increases. When it reaches 15 lb./sq. in., a bleed valve opens to exhaust the accumulated air, and the proportional band is simultaneously shifted downscale by blocking off the reset chamber. The controller output thus stays saturated during shut-down. On restart of the process, the reset chamber exhaust is shut and air returns to the reset chamber, reducing the controller output. The proportional band gradually shifts upwards and set point is reached without the usual cycling. It is emphasised that the new technique does not slow the response of the process; the approach to the set point is simply made more gradual on start-up. Normal process control remains unaffected subsequently.

Honeywell Controls, Ltd., Greenford, Middlesex.

#### Sheet Saw

A RECENT addition to the Darwins tool division range is the Steadfast sheet saw made by J. Stead & Co., Ltd. A 12 in. triangulated blade of Cobalterom special abrasion resisting steel is held in a polished alloy spine to which is fitted a shatterproof amber plastic handle. Two



blades of 14 and 24 teeth/in. are supplied and are available separately as replacements. This tool, which is obtainable from most hardware and tool dealers is intended to cut asbestos, plastics, wood, steel and other metals in a variety of forms, including corrugations, sheets, laminates, tubes, blocks, boards, etc.

J. Stead & Co., Ltd., Manor Works, Cricket Inn Road, Sheffield 2.

#### **Shore Hardness Testers**

SINCE its introduction over fifty years ago, the Shore Scleroscope has been widely used for hardness testing in industrial mass production and engineering. Two instruments are now marketed on behalf of the Shore Instrument and Manufacturing Company of New York by their sole agents for the United Kingdom and the Commonwealth (except Australia) Griffin & George (Sales), Ltd.

The new Shore Scleroscope Model C.2. is an improved, direct-reading instrument, simple and rapid in use for the

testing of thin metal sheets such as hardened steel (from 0·006 in. in thickness), cold rolled unannealed brass and steel (from 0·010 in.), and annealed sheets (from 0·015 in.). It is also suitable for the testing of chilled iron and forged rolls, gears, axles and other machined parts. It is claimed that the diamond hammer used leaves no impression visible to the naked eye even on strip with a mirror finish. Conversely, tests can be carried out on unpolished surfaces, which is not possible by certain other hardness testing methods. The Model C.2. may be used freehand and this enables inspection to be made anywhere along a production line and on material on machine tools, in presses and so forth. For continuous quality control at selected stations on the test bench the instrument can be held in clamping stands, swing-arm clamps or roll testing stands.

The Model D Scleroscope substitutes a dial and pointer for the reading of hardness numbers instead of the vertical scale behind the hammer tube. The dial, being also graduated in Brinell and Rockwell C hardness numbers, facilitates direct reading in any of the three scales desired. This instrument will test all the materials for which the Model C.2. is suitable, such as razor blades, large die blocks, shafting, crankshafts, machine tool ways, railway wagon wheels and so forth. It is not recommended, however, for use freehand. A selection of clamping devices is available. One operator can make over 1,000 tests per hour on a quick-moving production line, a rate claimed to be five times as fast as the speediest competitive device.

Griffin & George (Sales), Ltd., Ealing Road, Alperton, Wembley, Middlesex.

#### Gallium Arsenide

Johnson Matthey announce that they are now able to offer sample quantities of this intermetallic compound in polycrystalline form. It is also hoped that single-crystal material will be available shortly. Typical properties of polycrystalline gallium arsenide are given below:

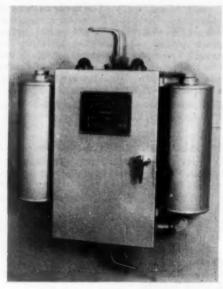
The material is available either as broken ingot or cut slices.

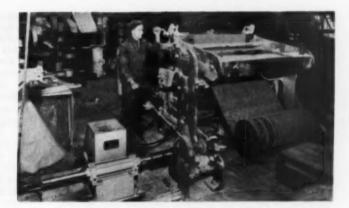
Johnson, Matthey & Co., Ltd., 73-83 Hatton Garden, London, E.C.1.

#### Small Air Dryer

ONLY recently introduced into this country from the United States, the B-6-A Budget Dryer is being manufactured by G.W.B. Furnaces, Ltd. This new dryer, developed by the Lectrodryer division of the McGraw-Edison Company, is suitable for applications requiring very dry air in small quantities—up to 20 cu. ft./min. A constant flow of dry air can readily be supplied to instrument controls, wave guides, small cubicles and other equipment, and since the Budget Dryer is easily moved and installed, it is especially well suited for pilot plant operations. When used with molecular sieve, the dryer can also be used for removing CO<sub>2</sub> from town's gas.

Once linked to the air supply and connected to any 230 V. A.C. supply, the Budget Dryer automatically





continues its cycle of adsorption and reactivation. If the electrical supply should fail, the air supply will not be interrupted, and instruments or other air-operated apparatus will continue to function. Automatic operation of the dryer is independent of operative pressures—from as low as 5 lb./sq. in. to as high as 150 lb./sq. in. The design calls for very little maintenance, as the valve mechanism moves only once every 3 hours, when the unit automatically changes adsorbers to return the previously reactivated unit to the drying circuit. The heater is extremely rugged, having been tested at overload voltage for several hours with no air flow—a far more rigorous condition than is likely to be

G.W.B. Furnaces, Ltd., Dibdale Works, Dudley, Worcs,

#### Rare Earth Metals

encountered in actual service.

Johnson Matthey announce that they are now able to supply scandium, yttrium, and most of the fourteen rare earth metals in sheet form in thicknesses down to  $0\cdot001$  in, and with a maximum width of 3 in. The sheet is available either cold worked or annealed. This is the latest development in the company's production of the metals of this group, which have previously been available in ingots and in some cases as wire of diameters down to  $0\cdot02$  in.

Johnson, Matthey & Co., Ltd., 73-83 Hatton Garden, London, E.C.1.

#### Edge or Line Guider

EDGE TRIM can be eliminated, speed of manufacture increased, and the quality of the finished product greatly improved by automatically positioning the edge of reel fed materials. The new Wadsworth-Lock edge or line guiding equipment consists basically of a sensing head and a completely independent, hydraulic ram control unit which is capable of adjusting very quickly in a lateral direction the position of reels weighing from a few pounds up to several tons. Alternatively, webs of

moving material can be positioned by using the hydraulic ram unit to operate a roller guider.

A number of edge or line sensing units are available, including a pneumatic detector and a fully transistorised electronic print line tracking unit which can guide a web at speeds up to 1,500 ft./min. from a continuous or broken line printed on the web. The pneumatic and electronic sensing heads as well as the hydraulic ram positioners have been carefully designed and engineered to ensure reliable trouble free operation under arduous industrial conditions.

A. M. Lock & Co., Ltd., Prudential Buildings, Oldham,

#### All-P.T.F.E. Pump and Coupler

Associated Electrical Industries, Ltd., have developed a pump whose working parts are composed entirely of PTFE (polytetrafluoroethylene) and a coupler for PTFE tubing. Chemical inertness and imperviousness to corrosive action are important properties of PTFE, and the pump will be particularly useful for pumping corrosive fluids, or liquids which must be protected from contaminating influences. It will therefore find a place for itself not only in chemical and other industrial processes, but also in the handling of certain liquid foods.

The new ÂEI pump is a diaphragm type and is self-priming. The vacuum-formed diaphragm and all the other PTFE parts in contact with the liquid being pumped are housed in a cast aluminium body which is botted directly to a capacitor-run A.C. geared motor. The motor gearbox provides a 7:1 step-down to give a diaphragm reciprocation speed of 200 cycles/min. Life of the diaphragm is more than 1,000 hours. Pump delivery at a 1 ft. head is 2 litres/min., and at the maximum working head of 15 ft. is 1·25 litres/min. Power consumption is 50 W. Overall dimensions of motor and pump are: length 10 in., width 5 in., height 5 in. Three models are available, to operate from 100/110, 200/220 and 230/250 V. 50 c./s. A.C. supplies.

Inlet and outlet ports of the pump are supplied "belled" to accept the new AEI PTFE pipe coupling (\( \frac{1}{2} \) in. size). The new AEI coupler is a spring-loaded telescopic unit which expands to grip the tubing inserted at each of its ends. It enables PTFE tubing to be joined so that only PTFE is exposed to fluids travelling in the tubes. The coupler, when used with tubing whose ends have been "belled," causes no restriction whatsoever

in the flow system; it is neat and easy to assemble and is capable of withstanding pressures up to 50 lb./sq. in., even at elevated temperatures (e.g.  $100^{\circ}$  C.). It is made in five sizes from  $\frac{3}{8}$  in. diameter to  $\frac{7}{8}$  in. in  $\frac{1}{8}$  in. steps. A simple tool for "belling" pipe ends can be supplied.

AEI Radio and Electronic Components Division, Fluorocarbons Section, 155 Charing Cross Road, London, W.C.2.

#### Mild Steel Welding Electrode

ELIMINATION of objectionable welding fumes, together with improved slag detachability are the main features of the new Diadem "Ruby" range of electrodes recently introduced by Cooper & Turner, Ltd. These mild steel electrodes are designed primarily for fast, high quality welds in the downhand position, although they are also suitable for vertical and overhead work. The Ruby range has full M.O.T., Lloyds and other official approvals. It complies with B.S. 639: 1952, and B.S. 1856 regarding mild steel welding requirements, and such other British Standards as B.S. 2642 covering the metal are welding of medium tensile weldable structural steel to B.S. 968. Careful matching of the core wire and covering make it particularly suited to the production of smooth dense welds of high strength, ductility and toughness by welders of only limited experience.

The Ruby range is designated E. 217 on the B.S. 1719 coding and E. 6012 on the A.W.S.-A.S.T.M. scale. The mechanical properties include a yield point of 24–28 tons/sq. in., an ultimate tensile strength of 29–34 tons/sq. in., and an Izod impact value of 50/70 ft. lb. The chemical analysis is carbon 0·08–0·10%, silicon 0·11–0·14%, manganese 0·55–0·65%, sulphur 0·025–0·035%, and phosphorus 0·015–0·025%. The electrodes are produced in a range of ten sizes, from 16 gauge to  $\frac{3}{8}$  in. and are supplied in 14 lb. or 7 lb. standard packs, according to size. The deposition time per foot of electrode at maximum current is 55 seconds for an 8 gauge electrode and 68 seconds for a 4 gauge electrode. Current values are 170 A. average and 190 A. maximum at 8 gauge and 290 A. average and 315 A. maximum at 4 gauge.

Cooper & Turner, Ltd., Vulcan Works, Vulcan Road, Sheffield, 9.

#### **Elapsed Time Meter**

A NEW elapsed time meter for recording the time a piece of equipment has been in use is being produced by English Electric. It can be applied to any equipment used intermittently and on which planned maintenance or a time log is required, e.g. pumps, ventilating and heating systems, valuable vacuum valves (X-ray transmitting tubes, etc.).

The meter is normally connected so that it is automatically switched on and off with the apparatus being metered. It can register up to 9999 9 hours in steps of 0·1 hour. The cyclometer register is driven by a self-starting, non-reversing, synchronous motor, through a train of gears. Operation is on A.C. supply in the voltage ranges 100–125, 200–250, and 400–440: the frequency can be 50 or 60 cycles.

The meter is available in two case types. The first, type H21M, is a 4 in. square, flush mounting, switchboard pattern case. The motor is mounted internally and two terminals are provided on the base plate to

connect the motor to the supply. The meter has a small revolving pointer to indicate that the motor is energised. The second is a  $2\frac{1}{2}$  in. or  $3\frac{1}{2}$  in., type S25 or S35, square moulded black plastic case designed primarily for panel mounting. The motor is mounted externally and connection to the supply is made via a pair of flexible leads. It is lubricated with a special grease for operation at extreme temperatures.

The English Electric Co., Ltd., English Electric House, Strand, London, W.C.2.

#### **British Chemical Standards**

BUREAU OF ANALYSED SAMPLES, LTD., announce the issue of the following new British Chemical and Spectrographic Standard Samples:—

B.C.S. No. 300—6% zinc aluminium alloy analysed for Cu, Mn, Si, Mg, Fe, Ti, Cr and Zn.

B.C.S. No. 304—10% aluminium bronze analysed for Cu, Zn, Ni, Fe, Mn, Si and Al.

B.C.S. No. 305—75% ferro-silicon analysed for P, Al. Ca and Si.

B.C.S. No. 306—0.4% carbon free-cutting steel analysed for Si, S, P, Mn and C.

These samples are only available in the finely divided form for chemical analysis.

S.S. Nos. 21–24—A series of low tungsten steels standardised for tungsten only, with tungsten contents ranging from 0.7% to 3.4%.

This series is supplied in the form of  $\frac{3}{4}$  in. diameter rods each 3 in. long for use as spectrographic standards, but is also available in the form of turnings for chemical analysis bearing the reference numbers B.C.S. 281–284.

Further details of these new samples are given in List No. 400R and the latest supplementary insert sheet. Bureau of Analysed Samples, Ltd., Newham Hall,

#### D.C./A.C. Inverter Units

Middlesbrough.

INVERTER UNITS for use in almost any situation where it is desired to operate, from direct current, electrical equipment normally requiring an alternating current mains supply, are now being produced by the electronic apparatus division of Associated Electrical Industries, Ltd. These D.C./A.C. units are now being made in three ratings, but a wider range is being developed. The first of the three units rated as at present was designed for the British Transport Commission to operate from 110 V. D.C. giving 230 V. A.C. at 1,200 c./s. and 240 W., and was for the fluorescent lighting of railway coaches. The second is a low priced unit of interest to farmers. It operates from any smooth 24 V. D.C. supply (such as tractor batteries), giving an output of 230 V. A.C., at 8 kc./s. and 40 W., and can be used for operating small fluorescent lighting installations. Commercial vehicle operators have also shown considerable interest in this unit for lighting in buses. Originally developed for I.C.I., for use as a power pack supply for shunting loco communication equipment, the third unit also operates from a 24 V. D.C. supply. The output, however, is 230 V. at 50 c./s. and 100 W., the unit thus being suitable for applications demanding larger power supply.

Associated Electrical Industries, Ltd., Crown House, Aldwych, London, W.C.2.

# CURRENT LITERATURE

#### **Book Notices**

#### PRINCIPLES OF METALLIC CORROSION

By J. P. Chilton. Monographs for Teachers: No. 4, 64 pp.. 34 diagrams, paper covers. The Royal Institute of Chemistry, 30 Russell Square, London, W.C.2. 6s.

For some years the council of the Royal Institute of Chemistry has considered it desirable to issue concise and authoritative accounts of selected well-defined topics in chemistry for the guidance of those who teach the subject at G.C.E. advanced level and above. It seemed that such guidance was particularly needed where there were difficulties in presenting a topic clearly in terms of simple fundamental principles and on a basis that would enable the pupil to go on to more advanced studies without having to "unlearn" what he had been taught at a more elementary stage.

The project, which received enthusiastic encouragement of the Science Masters' Association, was delayed owing to the lack of funds to support it, but the subsequent establishment of the Royal Institute of Chemistry Fund for the Development of Education in Chemistry made it possible to go ahead. As a start, three monographs were published on electrolysis, oxidation and reduction, and the extraction of metals, respectively, and as there was a wide demand for this kind of exposition it was decided to extend the series considerably.

Following an introduction in which the corrosion process is described as the complete reverse of that used for extraction of metals from their ores—and one, moreover, which tends to occur spontaneously because of the decrease in free energy attendant upon the conversion of metals into their compounds—the monograph proceeds to deal successively with corrosion in gaseous environment, corrosion in aqueous environment, prevention of corrosion, and some further corrosion phenomena. In this last section brief reference is made to certain effects of corrosion, including intergranular corrosion, stress corrosion and corrosion fatigue, in order to give a broader picture of the subject. Finally suggestions are made for further reading, headed, of course, by U. R. Evans' classics.

Though intended primarily for teachers of chemistry, the monograph will doubtless be of value to a wider readership, including more advanced students of chemistry and metallurgy, and, dare we add, those who once knew quite a bit about corrosion, but whose knowledge, through long non-use, is getting "rusty."

#### Trade Publications

"PLATINUM-IZED" titanium mesh anodes, trade-marked Platanium, which are reported to give solid platinum anode efficiency and quality performance at one-fifth the cost, are described in a new booklet by Sel-Rex Corporation, Nutley, New Jersey, U.S.A. The illustrated technical data on Platanium suggests uses and applications for this type of insoluble anode, which is said to be used extensively in applications ranging from platinum electroplating and electrochemical processing to refining catalysts and marine cathodic corrosion-protection.

The special diamond-configuration design affords maximum anode area while cutting down overall size at least 50%, according to the booklet. Basic anode sizes, shapes and types of anode hooks available—with corresponding prices—are given in the Platanium Anode Booklet, free on request from the company.

THE Summer issue of *The Magcan Tabloid*, eight page publication of the Magnesium Co. of Canada, Ltd., features an article describing the magnesium die casting process of The Outboard Marine Corporation of Canada, Ltd., who have installed equipment for automatic metering of the metal.

DATA for the engineer designer are provided in a new publication on the properties and applications of 'K' Monel, the nickel-copper alloy which combines the excellent corrosion-resisting properties of Monel alloy with mechanical properties as good as those of heattreated alloy steels. The publication, which consists mainly of charts and graphs on the physical and mechanical properties of the alloy, with sections on the available forms and applications, is obtainable free on request from the Publicity Department, Henry Wiggin & Co., Ltd. More copper alloys are extruded than fabricated by any other process. Although brass has been extruded since the beginning of the century, extrusion is a relative newcomer among metal fabrication processes. It has, however, become firmly established in the copper industry because of the ease with which most of the copper alloys can be formed when hot. It has been felt in the industry, therefore, that the story of copper alloy extrusions, simply told, must be of considerable interest to engineers, other industrial users, and technical students. A new addition to the existing wide range of C.D.A. publications-No. 59: Extruded Copper and Copper Alloy Products-now provides the essential basic information on the properties, manufacture and potentialities of the exceptionally wide range of shapes and sizes of rods and sections which is now available in a variety of copper-base metals. Many typical applications are illustrated, and by emphasising the technical and economic advantages it is hoped that this booklet will contribute to an even wider appreciation of the value and uses of extruded copper and copper alloy products. Comprising 72 pages and profusely illustrated, this publication is available free upon request to the Copper Development Association, 55 South Audley Street, London, W.1.

To read the June issue of *The Nickel Bulletin* is to realise the important role which nickel plays in practically every branch of industry. Subjects covered in the nickel section include the physical properties and the determination of the metal and the low-temperature characteristics of nickel/cadmium batteries, while in the following electrodeposition section attention is directed to literature on, *inter alia*, the corrosion-resistance of nickel/chromium electrodeposits, double layer - nickel plating processes, and industrial plating of screws and bolts. Items on the properties and applications of nonferrous alloys are followed by sections on nickel-iron magnetic alloys and nickel-containing cast irons (the latter including a reference to the requirements of an A.S.T.M. Specification covering austenitic cast irons).

Literature on structural steels is represented by abstracts indicating the scope and findings of work on the properties of high-strength steels, non-magnetisable steels. and, in particular, a 13% nickel steel for low-temperature applications. In the section concerned with heatand corrosion-resisting materials, the largest of the issue. attention is drawn to four compilations of published data on the physical and mechanical properties of representative nickel-containing high-temperature materials, to structural studies of nickel-base alloys, to work on the corrosion behaviour of aluminium-nickel alloys, and to papers reporting research on the mechanical properties, structural characteristics and corrosion-resistance of chromium-nickel stainless steels. An item of particular interest in this section is that covering a review of the economic considerations to be taken into account in selecting materials for marine applications.

"Hot galvanizing—the Process and the Product," a new booklet issued by the Hot Dip Galvanizers Association, is the second in a new series of technical booklets to be issued by the Association. It describes the hot galvanizing process and contains notes on the formation of alloy layers, spangle, corrosion resistance, fabricating and methods of finishing. It should prove to be of great value to technical students, engineers, architects and builders seeking basic information on galvanizing and its uses. Detailed information on all applications of hot galvanizing, may be obtained from the Hot Dip Galvanizers Association, 34 Berkeley Square, London, W.1. (Tel: GROsvenor 6636).

SINCE The General Electric Co., Ltd., marketed its motor control centre some three years ago, many improvements have been incorporated based on service experience. For example the range of control gear which can be accommodated has been extended to include starters up to 150 h.p. and switch-fuses up to 200 A. with no increase in the overall size of the cubicle. A duplex design for installations where floor space is at a premium has also been introduced. In the duplex cubicle the starter trays are mounted on both fronts with a saving of space of 6 in. over two single-fronted cubicles mounted backto-back. Technical Description No. 445 which covers this class of switchgear has been completely revised and much additional information added. Copies of this publication are obtainable from the company's Witton Publicity Department, Birmingham, 6.

The Cambridge Instrument Company have issued a new publication (List 325) describing their standard range of rare- and base-metal thermocouples for temperatures up to 1,500° C. In general, thermocouples are manufactured to customers' specifications and for particular applications, but certain types of thermocouple assemblies, which have been continuously in demand, are now produced as a standard range. The publication gives details of the factors governing the choice of suitable assemblies for widely differing applications, and describes a simple three-letter coding system which simplifies the ordering of stock assemblies and replacement parts.

BOOKLET S.7, on stock fan selection, is the fourth in a series dealing with equipment from the Tornado fan engineering range which is available from stock. The subject is the medium pressure, Type MP blower/exhauster which is stocked in five sizes for volumes from 20 to 3,000 c.f.m. at static pressures from 1.0 to 9.0 in.

w.g. As with other stock selection booklets published by Keith Blackman, this one is provided to enable the quick selection of the most suitable machine for a particular scheme, and restricts its contents to such essential information as dimension drawings, directions of discharge, motor positions and capacity tables.

LEAD is playing an increasing part in the field of atomics, and in a new brochure-" Lead for Radiation Protection "—Associated Lead (principal suppliers of lead shielding for the submarine Dreadnought and for the new C.E.G.B. laboratories at Berkelev) make available in concise form up-to-date information about lead in this role. This brochure gives much technical information on the mechanical properties of lead, the forms of lead shielding bricks available, and some of the alternative methods of providing reactor shielding. A section is also devoted to a new material called Densithene which is now being produced by Associated Lead and which is a composite lead/polythene product of particular importance in dealing with neutron and gamma-ray shielding. A special section deals with the use of lead in X-ray work. This booklet is available from Associated Lead Manufacturers, Ltd., Clements House, 14 Gresham Street, London, E.C.2.

Johnson Matthey & Co., Ltd., have issued a new data sheet describing their cobalt-platinum permanent magnet alloy Platinax II. This material is the latest development in magnetic alloys of the cobalt-platinum system, and with a BH $_{\rm max}$  of  $9\cdot 2\times 10^8$  gauss-oersteds is one of the most powerful permanent magnet materials known. Before its final heat treatment it can be rolled, drawn, or machined without difficulty. As well as general data previously included, the new publication gives information on the heat treatment process, on reversible permeability, and on behaviour at elevated temperatures. Copies of the data sheet and full particulars are available from the company at 73–83 Hatton Garden, London, E.C.1.

A NEW booklet has recently been produced by the Lead Development Association entitled "Production, Properties and Uses of Lead Cable Sheathing." Although the number of sheathing materials available for electric cables has increased in recent years, lead sheaths are easily manufactured, extremely reliable in service and relatively cheap. It was therefore felt that there was a need to supply accurate and up-to-date information on various aspects of the manufacture and use of lead for cable sheathing and this publication, the first in a new series, serves as a general introduction to the subject. More detailed information on all applications of lead may be obtained from the Lead Development Association, 34 Berkeley Square, London, W.1. (Tel: GROsvenor 8422).

The latest edition of Wiggin Nickel Alloys (No. 62) reviews applications of Wiggin high-nickel alloys in many of their specialised fields of application, the emphasis being on high-temperature materials specifically in the gas-turbine engine. This edition also includes articles on topics as diversant as the X15 hypersonic aeroplane and the corrosion-resistance of nickel/chromium plating, flexible expansion joints in Inconel alloy and boat-building using Monel alloy fasteners. The publication is obtainable free on request from the Publicity Department, Henry Wiggin & Co., Ltd.

# OF HORIZONTAL AND VERTICAL INTERNAL ELEMENT FURNACES BY

FOR VACUUM annealing brazing sintering

- TEMPERATURE RANGES-
- . SATURABLE REACTORS
- CHOICE OF GRAPHITE, OR
- COOLING EYSTEM
- "Up to 2400°C on some characteristics and tungsten aloments."

MODEL	CHARGE SPACE			
	Length	Width	Height	
0806	12"	8"	6"	
1209	18"	12"	9"	
1815	24"	18"	15"	
2418	30"	24"	18"	
3024	36"	30"	24"	
3630	48"	36"	30"	



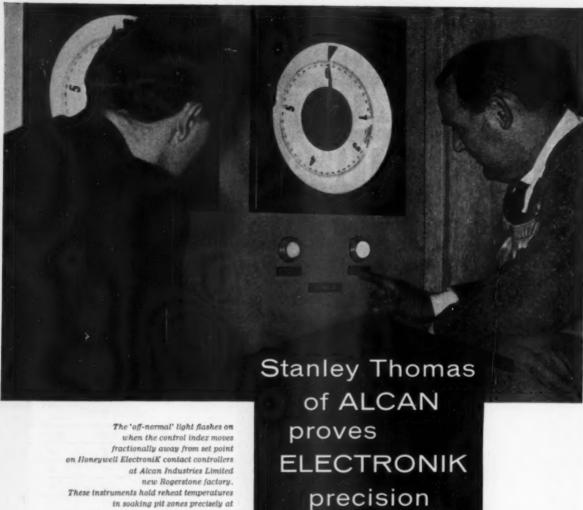
MODEL	CHARGE	SPACE
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POR VACUUM METALLURBICAL APPLICATIONS

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LEGPTHIN WORKS - OTTENS TO A WATFORD SY-PART WATFORD I MENTS. Tell I was like to the from the



new Rogerstone factory.

These instruments hold reheat temperatures in soaking pit zones precisely at set point, ±1°F.

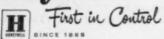
Electronik control is a proven guardian of aluminium quality in the Swindell-Dressler soaking pits which now 'feed' Europe's largest claving pits which now 'feed' Europe's largest claving

Electronik control is a proven guardian of aluminium quality in the Swindell-Dressler soaking pits which now 'feed' Europe's largest aluminium rolling mill. Temperature set point can be between 440°F and 630°F depending on the type of aluminium and the stage of heat treatment. It must be held to within  $\pm 1$ °F if aluminium excellence is to be kept consistent. Such precise control is easy for Stanley Thomas, ALCAN furnace reheat operator. Once he has set required temperature on the dial of Electronik controllers, the instruments take

over, ensure that the production specification is adhered to, unfailingly help the production of top class aluminium.

Automatic control of temperature and other variables is a Honeywell speciality throughout the metal industries. Honeywell engineers advise on conversion to automatic processing, devise complete control schemes utilising the broad range of Honeywell instrumentation, supervise their installation and keep them operating at peak efficiency by periodic maintenance.

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#### LABORATORY METHODS

MECHANICAL · CHEMICAL · PHYSICAL · METALLOGRAPHIC

INSTRUMENTS AND MATERIALS

SEPTEMBER, 1961

Vol. LXIV, No. 383

# A Mathematical Method for the Investigation of Inter-Element Effects in X-Ray Fluorescent Analyses

By H. J. Lucas-Tooth and B. J. Price

The Solartron Electronic Group Limited, Farnborough, Hampshire

Several investigators have tackled the problem of inter-element effects in X-ray fluorescent analyses from the theoretical aspect,\(^1\) whilst others have taken a more empirical approach and interpreted their practical results in terms of calibration curves and graphs.\(^2\) This paper suggests, from the broadest assumptions, what form these empirical formulæ are likely to take, and investigates how far these simple linear extrapolations can be used in practice.

A-RAY fluorescent analysis provides a means of determining accurately and rapidly the amounts of the various elements present in an alloy, and is particularly suitable for concentrations ranging from 0·1% to 100%. A complicating factor in the use of this method is the modification of fluorescent emission caused by inter-element effects. The intensity of the radiation from one element may be reduced by partial absorption by other elements in the matrix or, alternatively, its fluorescence may be enhanced by their presence. These effects are systematic and to a large extent predictable, and in the following pages a mathematic method for investigating the phenomena is presented.

#### **Theoretical Considerations**

Let us imagine a binary alloy containing two elements A and B, and let us imagine further that the fluorescent radiation of A does not excite B, and also that, for all wavelengths, the absorption coefficients of the two

elements are very similar. In these circumstances, admittedly unlikely in practice, the calibration curve of A (intensity of A radiation v. percentage of A present) will be a straight line, as shown in Fig. 1.

If, in the case of binary alloy AC, the element C strongly absorbs the A fluorescent radiation, a calibration curve of the type shown in Fig. 2 will result. The straight line calibration curve for A in alloy AB is also included in Fig. 2, the intensities for 0% and 100% A being identical in the two cases.

It will be seen from Fig. 2 that, in the case of ternary alloy ABC, containing, say, 60% A, the intensity of the A radiation can vary from  $I_1$  to  $I_2$ , depending on the relative concentrations of B and C in the remaining 40%.

#### Assumptions

At this stage it is proposed to make the following sweeping assumptions : the practical results will indicate their worth :—

(1) Apart from absorption effects, mutual enhance-

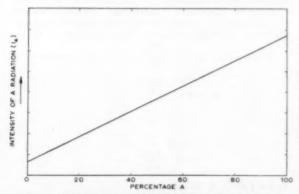


Fig. 1.—Calibration curve for element A in a binary alloy AB in which B does not absorb the fluorescent radiation of A.

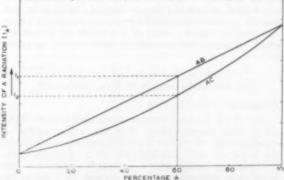


Fig. 2.—Calibration curve for element A in a binary alloy AC in which C absorbs the fluorescent radiation of A. The calibration curve for AB is repeated here.

ment also may occur. This will be regarded as negative absorption and assumed to obey the same

(2) The absorption of element A by a percentage of C (say  $P_c$ %) is linearly proportional to  $P_c$ . Thus, the actual departure, in terms of intensity, from the perfect AB binary calibration curves will be proportional to  $P_{\sigma} \times I_{A}$ .

The second assumption is not unreasonable: over small ranges, twice as much C might well be expected to have twice as great an effect on the A radiation. Also, the proportion of A radiation absorbed by a given amount of C is likely to stay approximately constant regardless of the amount of A radiation. Thus, for different  $I_A$ 's for a given  $P_c$ , the absorption is proportional to  $I_A$ .

#### The Form of the Equation

It is now possible to write down a projected interelement effect formula. First, it is important to define the notation. All the percentages will be written as P with a subscript immediately after it referring to the element—as in  $P_c$ . At a later stage, when different samples are considered, another subscript will be added denoting the sample numbers. Thus  $\hat{P}_{ss}$  refers to the percentage of the nth element in the mth sample. Similarly  $I_{sm}$  refers to the intensity of nth element in the mth sample. Using this notation, and taking the previous example,

$$P_{\scriptscriptstyle Am} = a_{\scriptscriptstyle A} + \beta_{\scriptscriptstyle A} I_{\scriptscriptstyle Am} + \gamma_{\scriptscriptstyle A} I_{\scriptscriptstyle Am} \, P_{\scriptscriptstyle Cm}$$

In practice we must consider more than one interfering element, and none of them is likely to be as similar to A as B was originally. In other words, to express the percentage of the nth element, in an alloy containing x elements, the formula which has been suggested so far is :-

$$P_{nn} = \alpha_n + \beta_n I_{nn} (1 + \Sigma_n \gamma_n, P_{nn})$$

 $P_{nm}=\alpha_n+\beta_n I_{nm}(1+\Sigma_z\gamma_{nz}P_{zm})$  This would be very inconvenient to use, as the percentages of the other elements appear in the right-hand side of the equation and they are, of course, related by similar equations, and hence solution of such equations would be involved.

The equation could be transformed into an expansion of this type:-

$$P_{nm} = a_n + \beta_n I_{nm} \ (\lambda + \sum_x \mu_x I_{xm} + \sum_x \nu_x I_{xm}^2 + \dots)$$
  
where the  $\lambda$ ,  $\mu$  and  $\nu$  coefficients are related to the constants  $a_n$ .  $\beta_n$  and  $\gamma_{nx}$ . As, however, we purposely neglected

any higher powers of  $I_s$  in our original assumptions, and also as v and succeeding terms can be shown to be composed of higher powers of  $\gamma_s$ 's which are themselves small, it is not unreasonable to write a final equation 88 :-

$$P_{nm} = a_n + I_{nm} (\kappa_o + \sum_z \kappa_{nm} I_{zm}) \dots (1)$$
  
( $\beta_n$  has been absorbed in the  $\kappa$  coefficients)

#### Evaluation of the Constant

Now if x is the total number of elements present in the alloy under consideration, then the total number of constants is x+2. Thus, if x+2 samples were available. whatever I's were obtained, the x+2 equations could be rigorously solved and a perfect fit obtained. This would not necessarily be the correct fit, due to inaccuracies in measuring the I's and the P's. In practice a much larger number of samples is used, and a leastsquares fit adopted. This can be very conveniently worked out on a computer as follows.

Define  $P_{nm}^{Form}$  as the  $P_{nm}$  calculated from the formula, and  $P_{nm}^{Obsm}$  as the chemical figure given on analyses from the sample. Consider the value A where

$$\triangle_{\scriptscriptstyle RM} = \begin{array}{c} P_{\scriptscriptstyle RM}^{\scriptscriptstyle Chem.} - P_{\scriptscriptstyle RM}^{\scriptscriptstyle Form.} \\ \hline I_{\scriptscriptstyle RM} \end{array}$$

This is a measure of the percentage departure of the percentage, as determined on the spectrometer, from the actual analytical figure. It is important that A should be expressed in this way, for the following reasons. Imagine two samples, one with  $P_n=5\%$  and the other with  $P_s=30\%$ . If we obtain X-ray percentages of 5.3% and 30.3%, respectively, we have in each case got an absolute error of 0.3%. However, the fractional error is worse in the 5% case, than in the 30% case. A better fit could be obtained if the 5% correction were improved, even at the slight expense of the 30% figure. To get the best fit possible, it is important to choose  $a_n$ ,  $\kappa_0$  and  $\kappa_1$ ,  $\kappa_2$ ...  $\kappa_x$ , so that  $\sum_{m} \triangle^2_{nm}$  is minimised. Thus, as

$$\triangle_{\it nm} = \frac{P_{\it nm}^{\it Chem} - P_{\it nm}^{\it Form}}{I_{\it nm}}$$

we can write

$$\triangle_{\rm nm} = \frac{P_{\rm nm}^{\rm Chom}}{I_{\rm nm}} \qquad \frac{\alpha_{\rm m}}{I_{\rm nm}} \ - \ \kappa_0 \ - \Sigma_z \kappa_{\rm nz} I_{zm} \label{eq:lambda}$$

Now define a matrix  $J_n$  as follows:—

a matrix P. as follows :-

and a matrix Kn as follows :x+2 columns

$$a_n, \kappa_1, \kappa_1, \kappa_2, \ldots, \kappa_s$$
 1 row

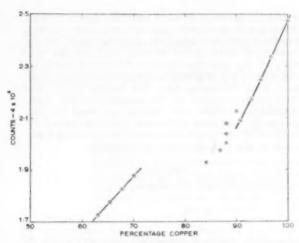


Fig. 3.—Graph showing percentage of copper plotted against counts. No corrections made.

In this notation formula (1) can be written:

It can be shown that  $\Sigma_m$   $\triangle_{nm}^2$  is minimised if  $\kappa$  is chosen to fulfil the following equation:

$$J_n^r P_n^{\text{Chem}} = J_n^r J_n \kappa_n \dots (3)$$

The procedure for evaluation is as follows. A large number of good samples are taken and their respective  $I_{nm}$ 's measured on a spectrometer. To work out the inter-element effect for the element "n," the equivalent  $J_n$  matrix is tabulated; similarly the  $P_n$  matrix is also tabulated. In the authors' case, these were then sent to the Ferranti Computer Centre at 21 Portland Place, London, W.1, which performed the necessary calculation and found the least-squares-fit the  $\kappa_n$  matrix.  $P_{nm}^{Pown}$  can be calculated for each sample and the results examined to see how good a fit is obtained.

#### **Practical Results**

It was decided, before applying this method to practical problems, that a problem with an unnaturally wide range of percentages would be undertaken to test the various assumptions. With this aim in mind a series of samples was prepared by the British Non-ferrous Metals Research Association, consisting of straight brasses, straight bronzes and some gun-metals. It would be extremely unlikely that, in practice, one would

TABLE I.—CHEMICAL ANALYSES OF COPPER ALLOY SAMPLES

Sample No.	Copper	Zinc	Tin
1	96-62	0	3-34
2	94-54	0	5-42
3	92-62	0	7 - 85
3 4	90-61	0	9-36
5	65-27	34-73	0
6 7	62-95	27-95	0
7	67-61	32-39	0
8 9	69-98	36-02	0
9	100-0	0	0
10	87.88	5-01	7-11
n	87-93	2-96	9-11
12	87-93	0.83	11-24
13	89-85	3-06	7:09
14	86-73	1-99	11.28
15	84.00	4-99	11.01

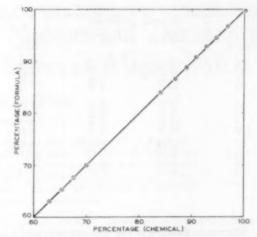


Fig. 4.—Graph showing the percentage of copper determined chemically plotted against the percentage obtained by application of the formula to the X-ray fluorescent results.

ever wish to fit these widely different alloys on the same calibration curve, but their accurate chemical analyses were readily available. Also, it was known that tin had a profound absorption on copper, and that tin radiation could further increase the copper fluorescent radiation by mutual enhancement.

The chemical figures for the alloys are listed in Table I, and the X-ray intensities, obtained on a Solartron automatic vacuum X-ray spectrometer, Type XZ,1030, are listed in Table II.

The copper intensity/percentage graph is plotted in Fig. 3 to show the serious nature of the inter-element effect. It is interesting to notice that in the worst case a 7% shift in copper percentage is required to bring these points on to one straight line. The following equation was given as the solution to the inter-element effect on copper, after the results had been computed in accordance with equation (1).

$$\begin{array}{l} P^{\rm Form}_{\rm Cu\,m} = \! 0 \cdot 78 + \! 10^{-4} \, I_{\rm Cu\,m} \, (27 \cdot 98 + \! 4 \cdot 84 \times 10^{-4} \, I_{\rm Cu\,m} - \! \\ 0 \cdot 64 \times 10^{-5} \, I_{\rm Sn\,m} + \! 1 \cdot 32 \times 10^{-4} \, I_{\rm Sn\,m}) \end{array}$$

Table III and Fig. 4 give a comparison of the closeness of the fit.

The root mean square difference is 0.13%. The reproducibility on the spectrometer for the copper

TABLE II .- X-RAY FLUORESCENT INTENSITIES OF COPPER ALLOYS

Sample No.	ICu	Iza	In
1	23,323	334	14,325
2	22,426	234	22,470
8	21,667	313	30,490
4	20,870	295	38,330
3	17,740	80,795	790
	17,247	53,618	778
7	18,244	47,799	785
8	18,750	44,680	840
9	24,724	349	800
10	30,771	7,778	29,030
11	90,376	4,559	87,175
12	20,031	1,659	45,018
13	21,220	4,845	28,515
14	19,738	3,028	45,215
15	19,361	7,252	44,985

TABLE III.—COMPARISON OF COPPER CONTENTS DETERMINED BY CHEMICAL ANALYSIS AND BY X-RAY FLUORESCENT ANALYSIS

Sample No.	P <sub>Cu m</sub>	P <sub>Cu m</sub>	Difference
1	96-62	96 - 78	+0.16
2	94.54	94-53	-0.01
3	92-63	92-85	+0.23
4	90-61	90-82	+0-21
5	65-27	65 - 27	0
6	62.95	63-03	+0.08
7	67-61	67-58	-0.03
8 9	69-98	69-94	-0.04
9	100.00	99-82	-0.18
10	87-88	87-64	-0.24
11	87-93	87-83	-0.10
12	87-93	87-94	+0.01
12 13 14	89-85	89 - 88	+0.03
34	86-73	86-60	-0-13
15	84-00	83-97	-0.03

intensity determination was 0.08% (standard deviation). It was considered that the chemical copper determination had been performed to a high accuracy and should not appreciably contribute to the error. It is worth noting, however, that although the three alloy types might give individually self-consistent chemical results, they might have some inter-group bias. The balance of the error must be apportioned to the simplicity of the inter-element corrections and, bearing in mind the unnecessarily large range attempted, the errors are satisfyingly small, and would, when used for correcting differences in any one alloy type, be negligible.

#### REFERENCES

Sherman, J., Spectrochemica Acta, 1955, 7, 286-306.
 Bareham and Fox, J. Inst. Met., 1969, 38, 344.

#### Stanton Balance for International Bureau

STANTON INSTRUMENTS, LTD., London, well-known manufacturers of precision, analytical, micro-chemical and thermo-recording balances, have recently supplied and installed a 10 kg. capacity high precision balance at the Bureau International des Poids & Mesures, Sevres, near Paris. The Bureau, which is the world's standards laboratory for those countries which adhere to the International Metric Convention, is re-equipping its Mass Section during the next few years. The 10 kg, balance is the first new high precision balance to be installed since 1910, and orders for further high precision balances have already been placed with Stanton Instruments, Ltd., for delivery during the next two years. The 10 kg. balance mentioned above is of the conventional threeknife edge design and is fitted with a special optical reading system. It will be used for the testing of larger weights and is expected to provide a precision of weighing of not less than 1 in 100 million.

#### Roto-Finishes Approved by A.I.D.

ROTO-FINISH, LTD., have received A.I.D. approval for their Cahill electroless nickel process and their Tridur AL chromate conversion process for aluminium under D.T.D. Specification Nos. 900/4728 and 900/4729, respectively.

The Cahill electroless nickel process is a method of plating by chemical reduction and produces a semi-bright, hard (V.P.N. 450/550) coating of nickel/phosphorus alloy (90–92% nickel — 8/10% phosphorus) on a variety of base metals; including steel, copper alloys, aluminium. By a suitable activation process it can be used for plating ceramics and phenolic mouldings.

The process is operated hot,  $88/93^{\circ}$  C., on a small scale in a glass or vitreous enamelled beaker, or on a larger scale in special glass lined tanks with electrothermal heating jackets. It requires no electric current or anodes; hence there is no problem in throwing power, and intricate and irregular shapes can be plated uniformly ( $\pm 10\%$  max. of the coating thickness). By a suitable heat treatment (1 hour at  $400^{\circ}$  C.) the hardness can be increased to 1,000 V.P.N. The process can be used for building up components machined undersize and for corrosion resistance, where it can replace expensive stainless or clad alloys. The exceptional hardness of the

alloy plate makes it particularly suitable for use on components where wear is a problem.

Tridur AL conversion treatment for aluminium and its alloys gives a high rate of corrosion resistance and good paint adhesion. Coatings are produced in 20 seconds to a maximum of 3 minutes, in a yellow to khaki colour which can be dyed with various coloured aniline dyes if required.

#### Gas Analysis Licence Agreement

It is announced that Elliott Brothers (London), Ltd., a member of the Elliott-Automation group, has negotiated with the Office National d'Etudes et de Re-cherches Aeronautiques (O.N.E.R.A.) of France a licence to manufacture and sell two gas analysis instruments of advanced design. The analysers are of the paramagnetic type, a principle used in both thermopneumatic and thermo-magnetic convection systems of measurement. The territory covered by the licence is, in general, world wide with some exceptions of which the principal are France and former French territories, the Benelux countries, North and South America and Japan. The new instruments are primarily intended for the determination of oxygen content in gaseous mixtures and have already found extensive application in the control of furnace atmospheres; catalyst protection, flue gas analysis and the control of atmospheric test rooms are further examples of their use.

British Furnaces, Ltd., Derby Road, Chesterfield, have recently received orders for gas carburising equipment to be installed in the new Bathgate factory of the British Motor Corporation. These orders cover six Super Allcase sealed quench furnaces, complete with fully automatic sequence control; one continuous pusher-tray type gas carburising furnace, together with the necessary "RX" endothermic atmosphere generating units; and various other items of auxiliary equipment.

Schloemann a.g. have been awarded a contract by the Hüttenwerk Oberhausen A.G. for the construction of a continuous rod mill, complete with auxiliaries. It is to be laid out to roll  $\frac{1}{6}\frac{3}{4}$  in. to  $\frac{5}{6}\frac{1}{4}$  in. diameter rod from  $\frac{3}{6}\frac{1}{4}$  in. square billets. The maximum delivery speed when producing  $\frac{1}{6}\frac{3}{4}$  in. rod will be 6,900 ft./min., and the rod mill will have a monthly output of about 30,000 tons.

# Simultaneous Determination of Niobium and Tantalum in Steel, Ferro-Niobium and Permanent Magnet Alloy

By L. Kidman and G. White

(English Steel Corporation, Ltd.)

A simple procedure is described for the simultaneous determination of niobium and tantalum in steels, ferro-niobium and permanent magnet alloy. The method depends on the solvent extraction of tantalum from the mixed oxides of tantalum and niobium obtained by precipitation with phenylarsonic acid from dilute hydrochloric acid solution. Trace amounts of tantalum are measured colorimetrically; niobium and amounts of tantalum greater than  $0\cdot 1\,\%$  are determined gravimetrically.

THE authors have previously described a method for the determination of trace amounts of tantalum in steels.¹ This method was extended to the determination of the higher percentages of tantalum in niobium-stabilised steels, but when applied to a series of experimental casts containing large amounts of both niobium and titanium, difficulty was experienced in the separation of tantalum from titanium. To overcome this difficulty, and to provide a means of the simultaneous determination of both niobium and tantalum, the authors have introduced solvent extraction. The result is a simple, quick and extremely versatile method.

Tantalum may be separated from niobium by an isobutyl methyl ketone extraction from hydrochloric acid medium. The amount of niobium extracted with the tantalum is a function of the concentrations of these acids in the aqueous layer, but if the solution is  $0.5~\mathrm{N}$  with respect to hydrochloric acid, a wide range of hydrofluoric acid concentrations can be tolerated without significant amounts of niobium being extracted with the tantalum. By means of this procedure, tantalum has been separated from niobium on a commercial scale, and this extraction has also been used in a method for determining tantalum in niobium pentoxide.

This solvent extraction procedure forms the basis of a new method for the determination of tantalum and niobium in steels, permanent magnet alloy and ferroniobium. The proposed method is suitable for the determination of a wide range of tantalum contents, including traces in steels, the larger amounts in niobium-stabilised steels, and the much higher contents in ferroniobium: it is also satisfactory for the determination of niobium in niobium-stabilised steels and in ferroniobium. After an initial separation with phenylarsonic acid¹ the mixed oxides are purified by solvent extraction. Traces of tantalum are determined by a colorimetric procedure and niobium by difference measurement; tantalum in ferro-niobium is more conveniently determined gravimetrically.

The efficiency of the separation of tantalum from niobium and titanium has been studied and the proposed method applied to steels of known tantalum content and to ferro-niobium.

#### Experimental

Recovery of Tantalum after the Separation Procedure

Aliquots of a standard tantalum solution to cover a range of from 0.5 to 2 mg. tantalum were transferred

to platinum capsules. These were evaporated to dryness and finally ignited at 800° C. The residues were dissolved in 5 ml. of 20% hydrofluoric acid solution and the solvent extraction procedure was carried out as described below. The tantalum in the extract was determined using the pyrogallol colour technique under conditions previously examined.<sup>1</sup>

Ta Added (mg.)	Ta Recovered (mg.)	Percentag Recovery
0.50	0.52	104
0.75	0.76	102
1.00	1.00	100
1.50	1.46	97
2.00	1.95	98

Recovery of Tantalum from Mixtures of Tantalum and Niobium

Varying aliquots of the standard solution were transferred to platinum capsules. To each aliquot was added 30 mg. of Matthey "Specpure" niobium pentoxide. The solutions were evaporated to dryness, ignited at 800° C., and the extraction procedure carried through. The aqueous layer and wash solution from each extraction were evaporated to fuming with sulphuric acid. After dilution the niobium was determined gravimetrically.

Ta Added (mg.)	$Nb_2O_5$ $Added$ $(mg.)$	Ta	Recovered (mg.)	$Nb_2O_5$ $Recovered$ $(mg.)$
0.25	30		0.25	30-5
0.50	30		0.50	30.0
0.75	30		0.73	30-6
1.00	90		0.08	90.5

Recovery of Tantalum in the Presence of Niobium and Titanium

To aliquots of tantalum solution were added 30 mg, of niobium pentoxide and titanium solution to give a range of titanium from 0·25 to 10 mg. After solvent extraction an aliquot of the final solution taken for colour development was acidified and examined for titanium by the hydrogen peroxide method. No titanium was detected, and in all cases separation was complete.

Ta Added (mg.)	$Nb_qO_5 \ Added \ (mg.)$	Ti Added (mg.)	Ta Recovered (mg.)
0.25	30	2	0.25
0.25	30	4	0-24
0.25	30	6	0.26
0.25	30	10	0.24

Ta Added (mg.)	$Nb_2O_5$ Added (mg.)	Ti Added (mg.)	Ta Recovered (mg.)
0.50	30	2	0.48
0.50	30	4	0.50
0.50	30	6	0.52
0.50	30	8	0.50
0.50	30	10	0.53
1.00	30	2	0.99
1.00	30	4	1.00
1.00	30	6	1.10
1.00	30	8	0.98
1.00	30	10	1.02

From the recoveries of tantalum and the fact that no colour was observed on the addition of peroxide to an acidified aliquot of the final solution, it may be concluded that tantalum is separated from titanium by this extraction procedure.

#### Recovery of Larger Amounts of Niobium and Tantalum

Varying amounts of Matthey "Specpure" niobium and tantalum pentoxides were fused with 10 g. of potassium bisulphate and, after extraction of the melt with water in an 800 ml. beaker, 80 mg. of pure iron were added as ferric chloride to simulate the conditions that would be encountered in the analysis of ferroniobium. The oxides were precipitated with phenylarsonic acid, filtered, ignited and weighed.

Wt. 
$$Nb_2O_5$$
 Taken  $(g.)$  Wt.  $Ta_2O_5$  Taken  $(g.)$  0 · 2255

Wt. Mixed Oxides Recovered (g.) 0.2505

After dissolution and solvent extraction the organic layer was evaporated to dryness in a platinum capsule and finally ignited at 800° C. for 10 minutes. The tantalum oxide recovered was then weighed. The aqueous extract and wash solution were evaporated to fuming with sulphuric acid. After dilution the niobium was determined gravimetrically on the solution.

Wt. 
$$Ta_2O_5$$
 Taken Originally (g.)  $0.0250$ 

Wt. Ta<sub>2</sub>O<sub>5</sub> Recovered After Separation (g.) 0.0245

> Wt.  $Nb_2O_5$  Taken Originally (g.) 0 · 2255

Wt. Nb<sub>2</sub>O<sub>5</sub> Recovered from Aqueous Layer (g.) 0·2240

These figures represent 98% recovery of the tantalum and over 99% recovery of the niobium.

#### Interference of Tin in the Presence of Iron

To 4 g, of pure iron dissolved in 40 ml. hydrochloric acid was added a standard solution of stannic chloride containing 0·1 g, tin. After adjusting the acid content of the solution, the phenylarsonic precipitation procedure was carried through. No precipitate was observed even after standing the solution at 90° C. for five hours.

#### Interference of Tin in the Presence of Niobium and Iron

50 mg. of niobium pentoxide were fused with 2 g. potassium bisulphate. The melt was leached with water and added to the solution containing 4 g. pure iron and

 $0\cdot 1$  g. tin. The phenylarsonic acid precipitation was carried through exactly as stated in the procedure. The ignited niobium was cooled and weighed.

It may be concluded that niobium is separated from at least 100 mg, of tin by the precipitation procedure.

#### Analysis of Ferro-Niobium

A standard sample of ferro-niobium, supplied by the London and Scandinavian Metallurgical Co., Ltd., has been analysed by the single precipitation method. The approximate percentage composition of this sample is:

Results obtained by the proposed method were:

The manufacturer's analysis quoted for another ferroniobium is:

Results obtained by the proposed method were:

	(a)	(b)
Niobium	66.8%	67.0%
Tantalum	6.72%	6.72%

Such analysis of ferro-niobium is performed comfortably in one working day.

#### Analysis of Steels and Permanent Magnet Alloys

In attempting to extend the scope of the method to cover the analysis of highly alloyed steels and permanent magnet alloys, it was found that a single precipitation of the oxides by phenylarsonic acid, followed by the organic extraction of tantalum, gave excellent results for the tantalum determination but the figures for niobium, obtained by difference, were slightly high due to the earth oxides being contaminated with traces of other elements, notably titanium and tungsten. These impurities were eliminated by re-precipitation provided that the sample contained not more than 0.25% tungsten. Using the proposed method, the niobium and tantalum contents of a number of complex steels and permanent magnet alloys were determined with the following results.

Sample	Nb (%)	Ta (%)	
A	1.25	0.098	
BCS 246	0.81	0.027	
BCS 261	0.67,	0.043	
BCS 266	1.09.	0.105	

The percentage composition of the test samples was:

Sample		Ni	$C_{\Gamma}$	Mo	W	Ti
A		8.8	20.6	0.50		0.41
BCS 246		12.0	19.0	2.9	0 - 2	-
BCS 261		13.08	17.20	-	-	-
BCS 266		13.3	-			0.09
Sampi	le	Co	Cu	Al	Nb + Ta	Ta
A		-	-		1.35	0.10
BCS 246			_	_	0.82	0.028
BCS 261		_		-	0.71	more
BCS 266		23.4	3 - 33	$7 \cdot 95$	1.21	-

An indication of the reproducibility of the method for tantalum is given in the following table.

Sample	Number of Determina-	Ta Present	Ta Found (%)			
	tions	(%)	Range	Mean		
A	3	0.100	0.097-0.100	0.098%		
BCS 246	5	0.028	0.026 - 0.028	0.027%		
		Method				

#### Apparatus

All optical densities were measured using the Spekker absorptiometer with mercury vapour lamp, Ilford 601 filters and H.503 heat filters. This instrument was chosen as, being the most widely used in steelworks laboratories, the method could be readily adopted for routine analysis.

#### Reagents

- (i) Wash Solution—Dissolve 0.5 g. of phenylarsonic acid and 1 g. of ammonium nitrate in hot water and dilute to 1 litre.
- (ii) 20% Hydrofluoric Acid—In a polythene measuring cylinder, dilute 100 ml. of conc. hydrofluoric acid to 500 ml. with water. Store in a polythene bottle.
- (iii) 10% Hydrochloric Acid—Dilute 50 ml. hydrochloric acid (S.G. 1·16) to 500 ml. with water.
- (iv) Boric Acid-A saturated solution.
- (v) Hydrogen Peroxide—100 vols. AnalaR hydrogen peroxide.
- (vi) Potassium Bisulphate—Fused potassium bisulphate.
- (vii) Phenylarsonic Acid
- (viii) 4% Ammonium Oxalate Solution—Dissolve 40 g. of AnalaR ammonium oxalate in hot water and dilute to 1 litre.
- (ix) 50% Pyrogallol Solution—Dissolve 50 g. of AnalaR crystalline pyrogallol in 100 ml. of cold 2% sulphuric acid using a mechanical stirrer (solution takes approximately one hour).
- (x) 25% Phosphoric Acid—Dilute 25 ml. of phosphoric acid (S.G. 1·75) to 100 ml.
- (xi) Zirconium Solution—Dissolve 0·3 g. of zirconium nitrate in 50 ml. of 20% hydrochloric acid and dilute to 100 ml.
- (xii) Standard Tantalum Solution—Fuse 0·1221 g. of Matthey "Specpure" tantalum pentoxide with 5 g. of potassium bisulphate. Extract in 4% ammonium oxalate solution. Dilute to 1,000 ml. with 4% ammonium oxalate. (1 ml.=0·1 mg. Ta or 0·001% Ta on a 10 g. sample weight.)
- (xiii) Iron Solution—Dissolve 0.5 g. of pure iron in a minimum of hydrochloric acid and oxidise with dropwise additions of nitric acid. Boil to expel nitrous fumes. Cool and dilute to 500 ml.

#### PROCEDURE 1—THE DETERMINATION OF TRACE AMOUNTS OF TANTALUM IN STEEL

Transfer 10 g. of sample (Note 1) to a 800 ml. squat beaker. Dissolve in 50 ml. of hydrochloric acid (S.G. 1·16) and oxidise by dropwise additions of nitric acid. Evaporate to dryness and bake lightly. Redissolve the residue in 40 ml. of hydrochloric acid (S.G. 1·16). Dilute to 100 ml. with water and filter through a paper pulp pad washing with hot 5% hydrochloric acid. Ignite the pad in a platinum dish. Add a few drops of 8% oxalic acid and approximately 1 ml. hydrofluoric acid, and then dry and ignite at 800° C. Fuse the residue with 2 g. potassium bisulphate, extract the melt with 10 ml. of

50% sulphuric acid and add to the filtrate.

Add 5 ml. of zirconium solution, 10 ml. of hydrogen peroxide (100 vols) and dilute to 500 ml. with hot water. Bring to the boil. Add phenylarsonic acid as a hot solution (1 g. dissolved in 20 ml. of hot water), stir well and add a small piece of paper pulp. Boil gently for 5 minutes. Set aside for 1 hour at 90° C.

Filter through a tight paper pulp pad, washing at least six times with hot wash solution. Ignite the precipitate in a porcelain dish at a temperature of 800° C. Cool and transfer to a platinum dish (*Note* 2).

To the mixed oxides add 5 ml. of hydrofluoric acid solution (20%) and heat gently until dissolved (Note 3).

When dissolved, cool, and pour the solution into a 100 ml. cylindrical separating funnel containing 1 ml. saturated boric acid solution. Rinse the platinum dish with 5 ml. of hydrochloric acid solution (10%) and add to the separating funnel. Add 10 ml. of iso-butyl methyl ketone and shake for 30 seconds. Allow the phases to separate and run off the aqueous layer into a second separating funnel containing I ml. boric acid. Return the organic layer to the original platinum capsule. Add a further 5 ml. of iso-butyl methyl ketone to the aqueous layer and repeat the extraction. Discard the aqueous layer, combine the two organic extracts and wash by shaking with a mixture of 5 ml. of hydrofluoric acid (20%) and 5 ml. of hydrochloric acid (10%) in a separating funnel containing 1 ml. boric acid. Run the organic layer into a clean 50 ml. platinum capsule and slowly evaporate to dryness. Ignite at 800° C. for 5 minutes and cool. Fuse the residue with 5 g. of potassium bisulphate and extract with 4% ammonium oxalate solution. Cool and dilute to 100 ml, with 4% ammonium oxalate solution.

Transfer a 50 ml. aliquot to a 125 ml. conical beaker. Add 4 ml. of 25% phosphoric acid and 20 ml. of ammonium oxalate solution. Adjust the pH of the solution to 2·1 with either phosphoric acid or ammonia using a sensitive pH meter (Note 4). Add 20 ml. of pyrogallol solution and dilute to 100 ml. with ammonium oxalate solution. Stand in a water bath at 26° C. (Note 5) for 15 minutes for full colour development. Measure the optical density immediately in 4 cm. cells using an absorptiometer with mercury vapour lamp and Ilford 601 filters (Note 6).

#### Calibration

To a series of 150 ml. conical flasks, transfer suitable volumes of standard tantalum solution to give contents of up to  $2\cdot 0$  mg. To each aliquot add 5 g. of potassium bisulphate and approximately 70 ml. of 4% ammonium oxalate solution. Warm until the solution is clear. Cool and dilute to 100 ml. in a calibrated flask. Transfer a 50 ml. aliquot to a 125 ml. beaker, add 4 ml. of 25% phosphoric acid and 20 ml. ammonium oxalate solution. Adjust the pH of the solution to 2·1 with either phosphoric acid or ammonia, using a sensitive pH meter, and add 20 ml. of pyrogallol solution. Using a graduated flask, dilute the solution to 100 ml. with 4% ammonium oxalate solution. (The pH of this solution lies between 1·8 and 2·0 if previously adjusted accurately.)

Develop the colour in a water bath at 26° C, for about 15 minutes. Measure the optical density immediately in 4 cm. cells using an absorptiometer with mercury vapour lamp and Ilford 601 filters. A linear relationship is obtained.

Notes

- (1) The method is suitable for tantalum contents up to 0.020%: it may be extended to cover tantalum contents up to 0.1% by reducing the sample weight and cell size.
- (2) Platinum dishes must not be used for the ignition; they can be seriously attacked, particularly at low temperatures, by the arsenical complex.
- (3) It was found that niobium-stabilised steels were difficult to dissolve in the hydrofluoric acid solution. The following technique gave solution quite simply and readily.

To the mixed oxides add 5 ml. of conc. hydrofluoric acid. Heat just below boiling till a clear solution is obtained and evaporate to incipient Redissolve in 5 ml. of the hydrofluoric acid solution (20%) and proceed with the extraction.

- (4) It has been found that, if the amounts of reagents added are controlled accurately, this step may be omitted.
- (5) Any temperature between 20° and 30° C. at which the solution can be accurately controlled is suitable. 26° C. was found to be most convenient for the conditions existing in these laboratories. Using higher temperatures it was found that the rate of fall of temperature was too rapid for accurate measurement of colour density.
- (a) Use the zero setting method (B.I.S.R.A. Special Report No. 55, p. 4, Method 1 b) with the reagent blank as compensating solution. Experience to date has revealed no detectable blank. (b) If a spectrophotometer be used for this measurement, the optimum wavelength is 400 mµ.

PROCEDURE 2.—THE DETERMINATION OF NIOBIUM AND TANTALUM IN FERRO-NIOBIUM

Weigh 0.25 g. of crushed alloy into a 50 ml. platinum capsule and fuse at a low temperature with 6 g. of potassium bisulphate until a clear melt is obtained. (If fusion of the last few milligrams proves difficult, cool and add 10 drops of sulphuric acid (S.G. 1.84) and carefully re-heat to a clear melt.)

Using hot water, extract the fusion in a 800 ml. tall beaker, add 40 ml. of hydrochloric acid (S.G. 1·16) and warm to dissolve. Add 10 ml. of 100 vols. hydrogen peroxide and dilute to 600 ml. with hot water. Bring to the boil, add phenylarsonic acid as a hot solution (1 g. dissolved in 20 ml. of hot water) and boil gently for five minutes. Set aside at 90° C. for one hour.

Filter through a tight pulp pad, washing at least six times with hot wash solution. Ignite the precipitate in a porcelain dish at a temperature exceeding 800° C. After ignition, transfer the precipitate to a weighed (A) platinum dish (Note 1) and treat with 1 ml. of 8% oxalic acid and 1 ml. of conc. hydrofluoric acid. Dry and ignite at 800° C. Cool and weigh (B). The difference between weights A and B is the weight of the combined oxides of niobium and tantalum.

Redissolve the oxides in 5 ml. of hydrofluoric acid solution (20%) and proceed with the organic extraction of the tantalum as described in Procedure 1.

Collect the final organic layer in a weighed (C) platinum dish and evaporate slowly to dryness. Ignite at 800° C.

for ten minutes. Cool and re-weigh (D). The difference between the weights C and D is the weight of tantalum pentoxide.

$$\%{
m Ta} = \frac{(D-C) \times 0.82 \times 100}{{
m Sample weight}}$$
 $\%{
m Nb} = \frac{(B-A)-(D-C) \times 0.7 \times 100}{{
m Sample weight}}$ 

Note

(1) Platinum dishes must not be used for the ignition: they can be seriously attacked, particularly at low temperatures, by the arsenical complex.

PROCEDURE 3.—THE DETERMINATION OF NIOBIUM AND IN NIOBIUM-STABILISED STEELS AND TANTALUM PERMANENT MAGNET ALLOYS

Dissolve 5 g. of steel (Note 1) in 40 ml. of hydrochloric acid and oxidise with nitric acid. Continue as in paragraphs 1 and 2 of Procedure 1, but omitting the use of the zirconium carrier.

Filter through a tight pulp pad, washing at least six times with hot wash solution. Transfer the precipitate and pad to the beaker and warm with 30 ml. of hydrochloric acid to redissolve. Dilute to 300 ml.. add 10 ml. of hydrogen peroxide (20 vols) and 5 ml. of iron solution and boil for three minutes. Reprecipitate using 0.5 g. of phenylarsonic acid dissolved in hot water. Stand for 1 hour at 90° C. Continue as in paragraph 3, in Procedure 1.

Weigh the mixed oxides before dissolving in hydrofluoric acid and continuing as from paragraph 4. From this weight and the percentage of tantalum determined colorimetrically, the percentage of niobium may be calculated.

Note

(1) The sample weight is governed by the amount of tantalum present. A sample weight of 5 g. is suitable for steels containing between 0.015 and 0.04% tantalum, a range which covers the usual niobium-stabilised steels.

#### Conclusion

A versatile and comparatively rapid method has been developed for the simultaneous determination of very wide ranges of niobium and tantalum. The method can be used for the determination of traces of tantalum in steels for nuclear engineering applications, or for the determination of niobium and tantalum in niobiumstabilised steels, permanent magnet alloys and the ferroniobium used in their manufacture.

#### Acknowledgments

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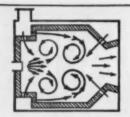


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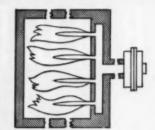
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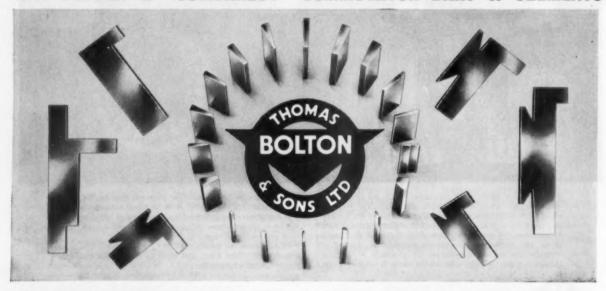
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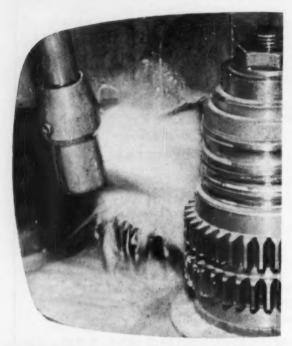
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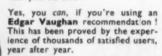
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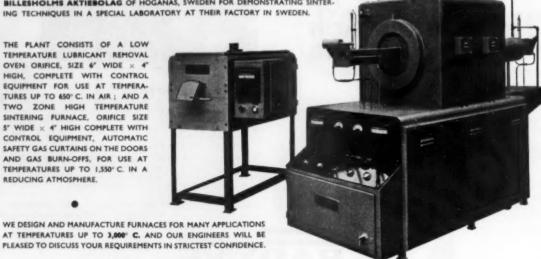
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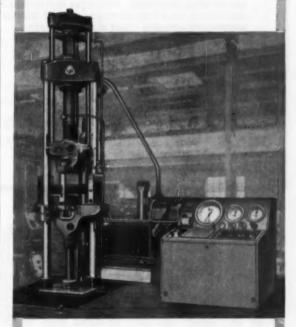
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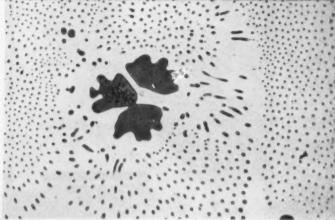
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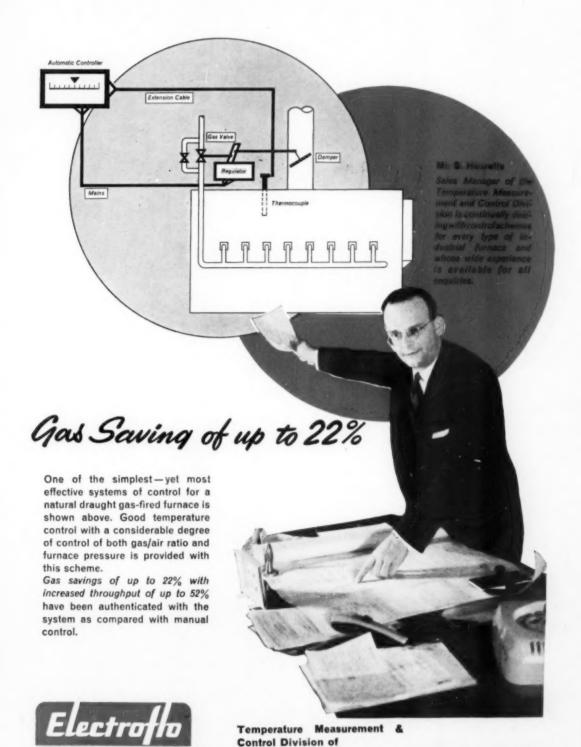
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